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Topics of the Month

Engineering of atomic piles

A CONSIDERABLE amount of chemical engineering is involved in the design and construction of nuclear reactors. Hitherto, however, because of the close secrecy surrounding Government-sponsored work on nuclear energy, it has been impossible to discuss the subject with anything approaching the freedom with which other scientific and technical topics are discussed. The folly of throwing the veil of secrecy over even the simplest details of nuclear energy has often been criticised by scientists here and abroad who have felt stifled by the hush-hush atmosphere thus generated. Their arguments and, no doubt, the series of leakages which has taken place since the war, have finally convinced the authorities that there is no point in withholding information which is already well known by all who have made it their business to find out. As a result, the Governments of Britain, Canada and the U.S. have decided to release more information on the design, construction and operation of low-power atomic piles. In this issue we publish in full one of the first documents to be released in accordance with the new policy—details of the Harwell Gleep, the first atomic pile to be built in Britain. Certain details of Bepo, the larger reactor at Harwell, will also be released at a later date.

Details of five other low-power reactors will be released under the scheme. Four are American and one Canadian: (1) The uranium and graphite reactor erected at the University of Chicago in 1942 and subsequently dismantled (the world's first nuclear reactor); (2) A modified version of this, now at the Argonne Laboratory, Chicago; (3) A uranium and heavy water reactor at Argonne; (4) An homogenous reactor using enriched uranium and ordinary water at Los Alamos, New Mexico; and (5) 'Zeep,' a heavy water and uranium reactor at Chalk River, Ontario.

None of the information to be released will have any value in the military application of atomic energy. It will be of great value, however, to chemical engineers and others who may be concerned with the development of industrial power from the atom and with its use in research. From brief preliminary details made available, of most general interest is the fact that the low-energy, thermal neutrons are captured in appreciable quantities by uranium-235 without causing fission. Earlier information has indicated that almost all thermal neutrons captured by the fissionable uranium-235 would result in fission. The technical articles will reveal, however, that a significant fraction of all thermal neutrons colliding with uranium-235 are captured to form uranium-236. Engineers designing uranium reactors must keep this fact in mind, since these captured neutrons are not available to continue the chain reaction.

A more precise value for the number of neutrons produced on fission of uranium-235 by thermal neutrons will also be reported in technical articles. This number hitherto has been quoted as being 'between 2 and 3.'

Other data to be released in technical articles include a description of the energy distribution of the neutrons released

instantaneously when uranium-235 is fissioned by thermal neutrons and a formula showing how 'lumping' natural uranium in low-power piles affects the capture of neutrons during their slowing-down process. All of these data are necessary in training nuclear reactor engineers and in designing low-power research reactors using uranium for their fuel.

Heavier demands for nitrogen

THE statistics of world nitrogen consumption and production are almost as important as indices of industrial and agricultural activity as are those relating to sulphuric acid. The annual report on the nitrogen industry published by Aikman Ltd., the London brokers, is thus a valuable service, particularly to the chemical and agricultural industries.

World consumption of nitrogen in 1950-51 is now expected to exceed the original estimate by at least 35,000 tons (all quantities in this note are in metric tons). Looking farther ahead, it looks as if production in 1951-52 will only just be enough to cover consumption (4,893,000), while in the following year demand (5,178,000) will exceed production (5,169,000). (Russian figures are not included in this estimate as they are unobtainable. Various private estimates, however, indicate an annual production of between 150,000 and 250,000. As a standard of comparison, U.K. production in 1949-50 was 358,000, in the U.S. 1,375,000.)

While there is no immediate shortage of nitrogen, it thus seems that demand will soon outstrip supply. Last year, for instance, production capacity was unexpectedly reduced. Firstly, the closing down of American war plants reduced production by 150,000 (however, since then one plant is said to have been reopened to produce nitrogen solely for war purposes). Secondly, the Korean war has caused the destruction of the 100,000 p.a. Konan plant. During the year also, there have been delays in the construction of European plants due to be in production between January 1952 and June 1953.

The increased consumption of nitrogen is due to heavier industrial demands, particularly in the U.S., Britain and Western Germany, and to increased fertiliser demands in the East.

Countries showing the biggest percentage increase in consumption in 1949-50 were Spain 70%, Denmark 31%, Sweden 30% and Holland 27% in Europe, and India 48%, China and Formosa 33%, Japan 20% and U.S. 5% in the rest of the world. Present estimates for 1950-51 point to substantial increases in Holland, France, Spain, Italy and Germany, the Near East, India, China, Formosa and the U.S., with a big reduction in Korea owing to the war. In other countries normal increases are expected.

In spite of heavier consumption, there was such a large carry-over of stocks from the previous year that between July and September 1950 there was a sharp reduction in the prices of synthetic nitrogen products, owing to producers competing to sell their stocks. This situation has now given way to a steady increase in prices as demands become heavier. On the other hand, the price of Chilean nitrate (in some cases 50% higher than that for synthetic nitrogen products) has remained steady and total production has been easily absorbed. There have been strikes in Chile which have increased costs, but with the new solar evaporation plant coming into production and more favourable exchange conditions recently approved by the Chilean Government, costs should again improve.

Gold ores yield uranium

URANIUM from South African gold mines, one of the world's biggest known sources, is to be made available to the British and American Governments. An agreement to this effect has been made between the three countries following recent talks held in Johannesburg. This agreement brings to a successful end several years of intensive research and development, in which all three countries have taken part, on the problem of making recovery of uranium from South African gold ores an economic proposition. Although there is not a large percentage of uranium in this ore, so much is mined that the total amount available will be relatively large, and the operation becomes economically possible because the gold 'tailings' (ore from which the gold has been extracted) are in most cases a waste product.

Four mining companies will be responsible for initial production. These are: West Rand Consolidated Mines Ltd., Daggafontein Mines Ltd., Blyvooruitzicht Gold Mining Co. Ltd., and Western Reefs Exploration & Development Co. Ltd.

Consideration will be given to the construction of additional uranium plants on other mines if required. Ultimately South Africa may become one of the world's most important uranium producers.

The Johannesburg negotiations were between representatives of the South African Atomic Energy Board, which includes leading members of the gold mining industry, and representatives of the United Kingdom and United States.

Design and construction of the necessary plant are proceeding as a matter of urgency.

Steels for pressure vessels

READERS of the report in our November issue of the Institution of Chemical Engineers' discussion on the new code for fusion-welded pressure vessels will recall that a document complementary to this standard was to be a series of standards for steels for vessels constructed to the code. This series has now been published as British Standards 1501-1506: 1950. The standards cover plates bars and sections, forgings, castings and bars for bolting material and comprise 50 specifications.

Having in mind the comprehensive character of this series and the possibility that further steels will be added to it from time to time, it was felt that some rational system of numbering should be adopted by means of which an indication could be provided of the type of steel, irrespective of the form. Appendix B explains in detail the system which has been adopted and indicates the basis on which it has been built.

It is realised that in providing a comprehensive series of all steels used in pressure-vessel construction it has been necessary to include certain steels already covered by existing British Standards. To have omitted these steels would have resulted in a document which would not have provided complete information, thereby greatly diminishing its value. Therefore, at the risk of repetition, specifications for these steels have been included, and Appendix C has been prepared to indicate any existing British Standards which are similar to those included in this series. The opportunity has also been taken to indicate the nearest equivalent American steel in either the A.S.T.M. or the A.I.S.I. series.

Copies of this publication can be obtained from the British Standards Institution, 24 Victoria Street, London, S.W.1, at 12s. 6d., post free.

Lord McGowan departs

THE news that Lord McGowan, chairman of Imperial Chemical Industries Ltd., was to retire on December 31, which was released just before Christmas, was not entirely unexpected. Lord McGowan is 76 and it was on medical advice that he decided to give up the leadership of one of the biggest industrial organisations in the world, after holding it for just 20 years. His must be among the longest associations with the chemical industry, for it is 60 years since he joined I.C.I.'s predecessors. In 1926, when I.C.I. was formed, he was chairman of Nobel Industries Ltd., one of the four companies which merged to form the new organisation. He became the first president and deputy chairman of the company on its incorporation and, four years later, on the death of the chairman, the first Lord Melchett, he assumed the position which he has just relinquished. Lord McGowan so completely identified himself with the great organisation which he helped so much to create, that it will be difficult to realise that he has departed from its active control. As *The Times* put it, the directors and high executives of I.C.I. who have for years been accustomed to looking to him for—and getting—guidance and decisions in difficult circumstances, will feel a great sense of loss. It is thus good to know that he has accepted the position of honorary president of the company as from January 1, so that the break will not be complete.

Lord McGowan's successor is Mr. John Rogers, O.B.E. Although he is not so well known as other I.C.I. deputy chairmen, his association with the company and its forerunners is almost as long as Lord McGowan's. Mr. Rogers first joined the organisation in 1899 and at the time of the merger was a director of Nobel Industries Ltd. He then became an I.C.I. director and in 1940 a deputy chairman. That he was unanimously elected to succeed his chief is a measure of the respect and regard he has earned from his colleagues and subordinates.

This change in the top management of I.C.I. is the third within a matter of weeks. It is only recently that Sir William Coates, a deputy chairman, resigned to devote himself to other business interests and on November 23 the board and industry generally mourned the unexpected and untimely death of another deputy chairman, Sir Frederick Bain.

Low-temperature grinding—a new process

A PROCESS recently developed by an American firm, Linde Air Products Co. of New York, makes a novel approach to the problem of powder-grinding very hard or heat-sensitive materials. Briefly, the normal mechanical grinding operation is accompanied by a spraying operation. The spray used is liquid nitrogen. The effect of this 'cold shower' treatment—and here 'cold' means -320°F.—is to bring the material rapidly to a state of maximum 'pulverisability.' No material needs to be brought to as low a temperature as that of the liquid nitrogen itself; the amount of cooling produced by spraying the liquid gas upon the material as it passes through a chamber between the feed hopper and the grinding mill is sufficient to bring about a significant increase in fragility. The choice of liquid nitrogen is, of course, based upon chemical rather than thermometric grounds. The gaseous nitrogen released as the liquid drops extract heat from the material is safely inert whatever the nature of the materials being pulverised.

There would seem to be several types of grinding problems to which the new method might be applied. First, the obvious case of the very hard material that is difficult to reduce to a powder by normal methods; the cold shower pre-grinding treatment may enable many such materials to be finely and rapidly ground in simple, light-duty mills. Second, there are the materials which melt or soften readily; they cannot be continuously ground, as overheating conditions are set up during pulverising. With such materials, grinding may become a tearing process, producing irregularly-shaped particles, but more often milling is brought to a standstill. Sufficient pre-cooling removes these difficulties. A third case is found in materials which are heat-sensitive, pharmaceuticals, substances containing vitamins and products which may lose aromatic components. Low-temperature milling will prevent losses or chemical changes that may occur as a result of the normal temperature rise of grinding processes. It is possible, too, that the predominating atmosphere of nitrogen will reduce the hazards of grinding explosive or readily oxidisable substances. However, the use of liquid nitrogen involves a sizeable addition to grinding costs. It is unlikely that the new method can ever be applicable to low-value materials. In such cases the use of cheaper cooling agents—possibly solid carbon dioxide or 'dry ice'—seems worthy of investigation when there are grinding difficulties.

Developing world iron and steel resources

WHILE there is a shortage of iron and steel in most industrialised countries, the known iron resources of the world are still very high. The ore deposits in the more populous under-developed countries are adequate to support a large iron and steel industry and in the smaller ones sufficient for economical mining, according to a report on 'World Iron Ore Resources and their Utilisation,' recently published by the United Nations Secretariat. This report is the first of a series of studies prepared by the Economic Stability and Development Division of the Economic Affairs Department, following a resolution of the Economic and Social Council emphasising the need for better utilisation of world resources in order to promote a higher standard of living throughout the world and particularly in the under-developed countries.

Lack of good-quality coking coal is often a serious problem in these countries, but a worse handicap is the limited local demand for iron and steel products. Nevertheless the establishment of a steel industry can itself contribute to the expansion of related industries and a more diversified economic development. In some of the larger under-developed countries the beginnings of a steel industry exist, but none of these has markets which are large enough to sustain plants producing all the different types of steel products, whereas in a few countries the demand already exceeds production. Various methods for reducing the requirements of coking coal or eliminating its use altogether by substitution of other coals, oil, natural gas or electric power are used to a limited extent in some countries, and such methods are likely to find wider application in under-developed countries.

In Africa iron-ore deposits are extensive in many regions and some are already being exploited. The mills are often rather small and the equipment not of the most modern design. However, this is not always economically unfavourable in under-developed countries, as labour costs are much lower than capital costs in comparison with industrialised countries.

An annual production of 20,000,000 tons of iron and steel, more than ten times the present levels, appears to be

feasible in China and India on the basis of their iron ore and coking coal reserves, according to the report, which adds that this development could be helped by an exchange of ores and coking coals within these areas. Latin America also possesses enormous reserves of high-grade iron ores but, with the exception of Mexico, has little coking coal of high quality well located in relation to ores or markets.

Whether these vast untapped resources are eventually exploited will to a large extent depend on the initiative of the industrialised countries of the world to provide the necessary technological advice and technically trained men. Due to the inherent disadvantages associated with many of the deposits, it is probable that the efficiency of the iron and steel production will initially not be as high as in the industrialised countries, although the quantity, quality and location of the reserves are advantageous for establishing or expanding a steel industry. As a consequence, these new steel industries will probably need some kind of protective measures against imports similar to those that the steel industries of the industrialised countries had in comparable stages of development and still enjoy.

Salt and its significance in India

IT would not be surprising if much of the low productivity of the average Indian worker is due to deficiency of salt, writes Sri E. B. Tisseverasinghe in his monograph, 'India and the Salt Industry,' where he suggests that it might be better to subsidise salt or even give it away to achieve greater industrial productivity. It is estimated that the present annual per capita consumption of salt in India is 14 lb., compared with 20 lb., the quantity required for normal health, leaving India with a total present requirement of 2,500,000 tons p.a. for domestic and trade purposes. This figure will, it is added, be increased to 4,000,000 tons in 10 to 15 years' time, due to rapidly increasing population and to improved standards of living.

Salt production in India could, however, be increased within a short period and later self-sufficiency could be attained, according to the recent report of the Committee of Salt Experts constituted by the Government of India in 1948.

There is at present only one modern salt works and by-products plant in India; this is located at Mithapur and owned by Tata Chemicals Ltd. (see INTERNATIONAL CHEMICAL ENGINEERING, September 1950, p. 409). Such developments are exceptional in India and the salt industry remains in a recumbent and primitive condition, uninfluenced by modern technological developments. The industry is in dire need of technical aid and the time has come for giving serious attention to improving the quality of Indian salt, which in many areas is sub-standard and unfit for human consumption.

The unique resources of the inland lakes of Rajputana have so far received only scanty attention and yet they represent vast chemical reservoirs constantly replenished by streams flowing over salt-bearing beds. The salt formed in the Rann of Kutch and distributed over the catchment area of the lakes by wind is dissolved by rain water and carried to lakes, whose salt contents are thus continuously enriched. The resources of some of these lakes are inexhaustible. Like the salt lakes of California and Nebraska, the Great Salt Lake of Utah and the Dead Sea, the Rajputana desert lakes await full exploitation. Moreover, climatic conditions prevailing in the lakes areas are very suitable for solar evaporation production methods.

By supplying proper conditions for the crystallisation of marine salts, it is possible to separate the valuable salts from those of lesser value and individual salts from their mixed solutions. The waters of Sambhar and Didwana lakes are rich sources of sodium sulphate, sodium carbonate and sodium bicarbonate. For every ton of common salt recovered from Didwana Lake, it is possible to recover $\frac{1}{4}$ to $\frac{1}{3}$ ton of sodium sulphate. The recovery of all these by-products would not only help to build up a flourishing chemical industry but, at the same time, improve the quality of salt produced and lower the costs of production.

In spite of huge salt resources and favourable climatic conditions, development of the Indian salt industry poses many problems. The Salt Research Committee of the Council of Scientific and Industrial Research has already started several practical research projects. The Committee of Salt Experts has recommended the establishment of model factories for undertaking large-scale development programmes in the more important salt-producing regions. If this recommendation is accepted and implemented, new facilities would become available for the commercial application of the results of research sponsored by the Council of Scientific and Industrial Research. The report ends by urging a determined effort by all concerned to put available knowledge into action and to transform plans into plants and processes.

One feels that, even if only a fraction of the enthusiasm of the writers of the report is translated into deeds and, of course, if the necessary technical skill and plant is made available, then a good beginning will have been made to establishing salt as a basic Indian chemical industry. The potential in only ten years' time, again to quote Tisseverasinghe, is some 10,000,000 tons of salt p.a.

The Rutherford Memorial

LORD Rutherford of Nelson was the greatest physical scientist of his time. His discoveries have led us to almost all we know about the structure of atoms, and the development of atomic energy and its by-products are the practical results of his work. During his lifetime his prodigious genius received the full recognition of his fellow scientists and of the world. In 1903 he was elected F.R.S., in 1905 he was awarded the Rumford Medal of the Royal Society, in 1909 he received the Nobel Prize for Chemistry, in 1914 he was knighted, in 1919 he succeeded J. J. Thomson as Cavendish Professor of Experimental Physics at Cambridge, from 1925 to 1930 he was P.R.S., and in 1931, six years before his death, he was created a baron.

Rutherford was not purely an academic scientist. Industry has cause to be grateful for his wise guidance to the Government on the extension of the scientific method in industry given during his chairmanship of the Advisory Council of the D.S.I.R.

Interpreting a widespread wish, the Royal Society now propose to perpetuate Rutherford's memory with a memorial taking two forms: Rutherford Scholarships tenable for three years to be awarded to British Commonwealth post-graduate students; and a Rutherford Memorial Lecture to be delivered in centres in the Commonwealth overseas. A substantial endowment fund will be needed for this purpose, and an appeal for contributions was launched by Sir Robert Robinson, as one of his last acts as president of the Royal Society. Contributions should be sent to the Rutherford Memorial Committee, the Royal Society, Burlington House, London, W.1.

Production and Applications of Mineral Wool

By A. M. MacGregor

(Stillite Products Ltd., Stillington, Stockton-on-Tees)

The manufacture of mineral wool developed in the second half of the last century as the result of efforts to find profitable uses for blast furnace slag. Slag is still used to manufacture mineral wool, the process involving the melting of slag at high temperatures and its fiberising with steam. In this article,* the author outlines this process and describes the production of the different forms of mineral wool. Finally, he reviews its many uses as an insulating medium and as an air filter for, among others, chemical works.

ALTHOUGH it is only within the last 30 years that mineral wool has achieved prominence in industry, it has been known for more than 100 years.

The first known production of a mineral wool was in Wales in 1840, where it was produced from slag. A short time later, production was started in Germany, and a mineral wool company has been in operation in New Jersey, U.S.A., since 1875.

Discovery of the property of slag giving a fibrous product when shredded with steam is said to have been accidental. The story goes that a ladle of molten slag was upset near a leaking steam pipe, and the slag entrained in the steam was carried away and deposited around the works as fibre. In any case, the utilisation of slag had been a problem which, for a long time, was occupying the minds of all blast furnace operators, as slag heaps were growing at the rate of 15,000,000 to 18,000,000 tons p.a.

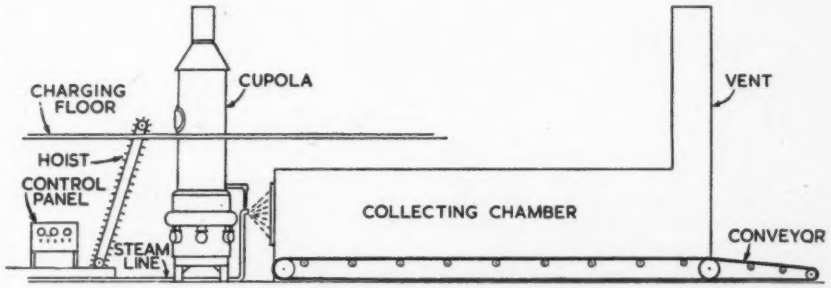
The main outlet for the use of slag was in road-making, for which it is still used. It was used in the manufacture of cements and, of course, even to the present day it is the base used by many mineral wool manufacturers.

Naturally occurring mineral wool is very rare. There is only one known source, in the Kilaeau crater, Hawaii, where it is known as ' Pele's Hair.' This is formed by the ejection of steam through fluid basalt lava from the crater. Otherwise, mineral wool is a manufactured material.

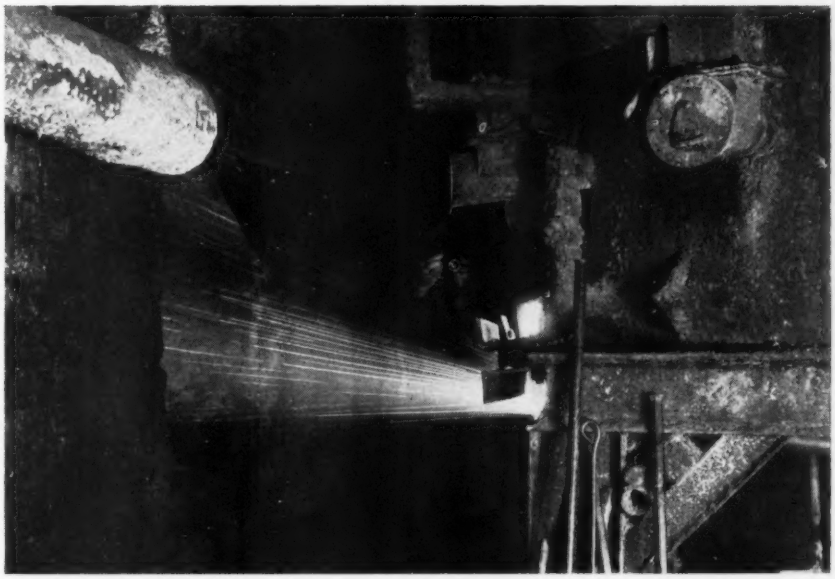
In the late 19th century it aroused considerable interest, work on the material being carried out by Charles Wood of Middlesbrough, who called it ' slag-wool ' or ' silicate-cotton.'

The fire-proofing qualities were fully realised and demonstrated, while the insulating properties, acoustic and sound-deadening advantages over other materials made it a popular material.

*Paper read at a meeting on October 12, 1950, of the Newcastle Branch of the Incorporated Plant Engineers.



Layout of plant for mineral wool manufacture.



Beginning of the fiberising process when the jet of steam strikes the molten mineral. The man in the centre background is measuring the temperature of the molten mineral.

It was also thought to be an antiseptic, and one writer opined that it had a future for medical purposes (Gilbert Redgrave, *J. Soc. Arts*, 1890, p. 225). As a matter of fact, it was used for cholera belts for use in warm climates and for chest protectors.

The development of mineral wool from either slag or rocks gravitated to America, where the potential market was much greater owing to the need there for a cheap and serviceable insulation for dwelling-houses and other buildings, and also due to

the growth there of mass production technique in the manufacture of domestic appliances.

Production in America expanded rapidly when the pneumatic method of installing it in buildings was perfected in 1930 and, in the ten years between 1928 and 1938, production increased about six-fold.

In this country, production has steadily risen according to the industrial and capital expenditure demand, but the advantage offered to building insulation has not yet been realised, which is rather puzzling, for mineral wool insulation, 1½ in. thick, gives the same protection as 3 ft. of brickwork against the extremes of weather.

Nature of the fibre

When a liquid is apparently at rest, this actual state of rest is only true of the bulk, the molecules of the liquid having some movement relative to one another. As the liquid cools, the molecules take up positions giving a definite spatial relationship with one another, and when the liquid is frozen the molecules have formed a definite geometrical pattern which is characteristic for any one compound. These patterns are, of course, crystals.

Crystalline solids have common properties, of which the following are noteworthy. They (*a*) have a sharp melting point; (*b*) are generally brittle or friable; and (*c*) exhibit different properties in different directions, for example crystals will usually conduct heat and electricity better in one direction than in another, and tensile strength depends upon the direction across which it is measured.

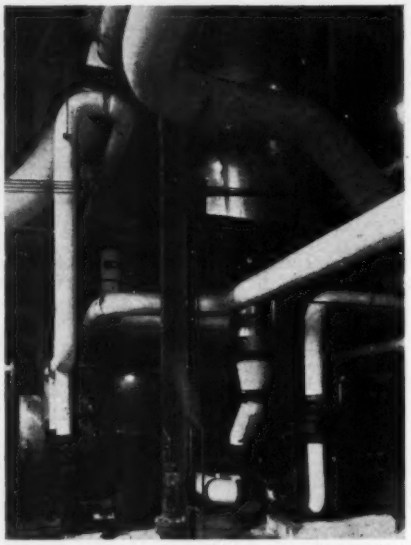
Some liquids, particularly viscous ones, do not crystallise out on cooling. It is easy to see why, when the movement of the molecules during the cooling process is followed. In a viscous liquid this movement is restricted by internal friction and, with further cooling, the movement is reduced as the viscosity increases. Eventually, the friction is so great that the molecular motion is brought to zero and the liquid will no longer exhibit any tendency to flow. In other words, the solid state has been reached before the molecules have had time to migrate to the positions necessary to give a crystalline solid, so that in this case there is a random arrangement of molecules. This solid state is called the amorphous or super-cooled liquid condition of matter. Amorphous is the more accurate term.

The characteristics of an amorphous solid, therefore, are different from those of a crystalline solid, in that: (*a*) they have no sharp melting point, but have a plastic range before becoming true liquids; (*b*) they do exhibit a degree of resiliency; and (*c*) physical properties, such as tensile strength and thermal conductivity, are independent of direction.

It can be readily understood that a crystalline fibre would be hard to produce, since the ratio length: breadth: thickness is constant for any one compound, and to produce a fibrous material from liquid crystallising out would require crystals to have a ratio of a very high number for the length and very small numbers for the breadth and thickness. There is no mineral with these properties.

A solid in the amorphous condition can, of course, be built up in any way desired, since the molecular arrangement in the structure is entirely at random. Therefore, for the production of mineral fibres, the mineral must be so chosen that it exhibits a tendency, when cooling, to pass into the amorphous state. This property is enhanced in the fiberising process, since the fibres have a high surface area: volume ratio and, therefore, cool very quickly, permitting even less time for crystallisation than when cooling in bulk.

Two other factors must also be considered. The mineral should have a high specific heat, so that it retains its heat as long as possible when fiberised, and it should have a 'long' plastic range, *i.e.* it should have a wide range between the temperature at which it first softens and the point at which it becomes really fluid. Both of these conditions promote the production of long, fine fibres.



[*Photo: Petrochemicals Ltd.*

The pipelines of this boiler feed-water de-aerator are insulated with mineral wool moulded sections covered with hard-setting composition. Semi-rigid slabs between aluminium sheets are used to insulate the de-aerator.

Manufacture

The plant unit consists of stockyard, conveyor or hoist to the charging floor, melting cupola and fiberising chamber.

The cupola is of the vertical type, surrounded by a water jacket. There is a controlled air blast fed into the cupola through a series of tuyeres. At the bottom of the cupola there is a notch through which the molten mineral is continuously tapped off. Suspended below the notch is a V-shaped nozzle of special design, through which steam issues at 100 lb./sq.in. pressure. This nozzle is so placed that the molten mineral drops on to the apex of the V. The fibres are blown into a collecting chamber, where they settle and are removed by a conveyor forming the bottom enclosure to the chamber.

In practice, the production system is as follows:

The mineral is quarried and stocked in bins according to its chemical composition. It is selected from the bins and crushed to a suitable size, between 4 and 8 in. across any one dimension. Hard foundry coke is used for smelting; a layer of this is added to the cupola, followed by the mineral charge. As the latter makes its way down the cupola, it is dried by the hot gases of combustion rising up through the charge. If any carbonate is present, dissociation takes place. As the combustion zone is approached, in the region of the tuyeres, the coke burns more vigorously and is reduced in volume, allowing the mineral to fall into the hot zone which is maintained at about 1,500°C. The mineral is fused and trickles down through the burning coke which, by now, is much diminished, and meets with the remainder of the previous charge. As the coke disappears, except for the ash, fusion of the charge is complete, and a well of molten mineral is obtained at the bottom of the cupola. The mineral flows through the notch on to an arrangement which divides it into a number of identical streams. The steam issuing from the V-shaped nozzle is the fiberising medium. The mineral drops on to the apex of the steam stream which is travelling at an initial velocity of some 1,000 ft./sec. An atomising process takes place—it is disintegrated into fine droplets which are carried into the collecting chamber by the force of the steam. During their flight, these droplets are attenuated into long, fine fibres by the air resistance to their flight. This is a spectacular part of the process. On looking into the chamber, the fibres can be seen as myriad red-hot streaks being rapidly carried along the chamber. The length of fibre produced from 1 lb. of mineral is of the order of 100,000 miles, and it is estimated that the daily output of fibre would girdle the earth's orbit round the sun ten times. Over 80% of these fibres are less than 6 microns diameter, the average being about 4 microns.

It will be realised that the conditions inside the collecting chamber are somewhat turbulent, despite precautions being taken to streamline the flow as much as possible. Even though the fibres have a degree of resiliency, collisions between fibres which take place would break them up unless additional protection to the fibre is offered. Therefore, a dressing is applied to the fibres which not only protects them but fulfils two other functions as well. The dressing applied is an adhesive which serves to bond the fibres into a blanket,

while the other function is a most important one which will be explained in more detail.

The main factor which governs the production of high-quality, fine fibres is the relationship between the viscosity of the fiberising medium and the medium being fiberised, that is the steam and the molten mineral respectively. The ratio of their viscosities should, as far as we know, be 1:1. This, of course, is impossible, since no gas has a viscosity approaching that of a liquid.

However, by entraining in the steam a chemical which will suspend in the steam, a higher viscosity is obtained, which increases the ratio of the viscosities and which helps to produce a fine quality of fibre. The types of chemicals chosen are organic adhesives of high molecular weight. In addition, bituminous matter is introduced through the medium of the steam, to impart water-shedding properties to the fibres.

These dressings come out of suspension in the steam when the latter has expanded beyond the point where it can retain a suspension and, being in a very fine state of division, it readily deposits on the fibres when the latter are cool enough to condense the vapour sheath which surrounds them. In this way, the individual fibres are given their dressing. The fibres are treated in the same manner with synthetic resins, oils, waxes and modified naturally-occurring resins, according to the requirements of production.

We now have the fibres, suitably treated, in the collecting chamber. They settle rapidly, for, although the total momentum is large, the momentum of each individual fibre is small, and they decelerate rapidly. In fact, a 60-ft. chamber is of sufficient length to accommodate the fiberising process—a remarkable fact, considering the mineral is struck by steam travelling initially at about 1,000 ft./sec.

After the fibres have settled, they are withdrawn from the chamber, in the form of a continuous blanket, by the conveyor unit, to be disposed of according to the requirements of the moment.

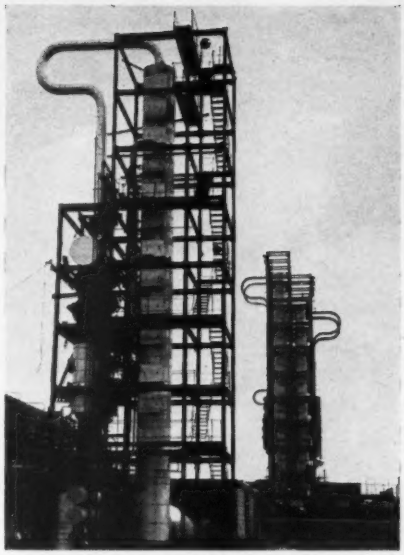
Processing the fibre

The products have been designed, in collaboration with industrial users, to meet the maximum number of applications.

Felted mineral wool is the fibre picked straight from the chamber conveyor. It requires no further processing before application. To permit easier application, especially for filling and packing cavities, a pelleted form of mineral wool is also available.

The mineral in pelleted form is produced from the wool from the chamber, by forcing it through a grid designed to give pellets of $\frac{3}{8}$, $\frac{1}{2}$, $\frac{3}{4}$ or 1 in. size. The broken wool is collected at one end of a vibrating screen, on which it is graded and turned into pellets.

Semi-rigid board insulation. A large proportion of the output of mineral wool is processed into boards by a specially designed unit. Material of between $3\frac{1}{2}$ and 21 lb./cu.ft. density is made on this machine by varying the conveyor speed through the system and the binder deposited on the fibre.



[Photo: Petrochemicals Ltd.

The columns of this azeotropic distillation unit have mineral wool lags and the manhole covers are wire mesh mattresses with sheet aluminium cladding.

To produce this material, the conveyor running along the chamber is extended to include compression devices; an oven, to dry out and bake the adhesive used; and a series of cutting saws to dimension the board as required.

The blanket, as it emerges from the collecting chamber, is conveyed in a moist state through a compressing belt adjusted to give the required thickness of material, then on to a series of rollers which complete the compression and tidy the surface of the material. The blanket is transferred by conveyor into a drying oven about 100 ft. long which removes the moisture and bakes up the adhesive. Emerging from the drying oven, the blanket, now termed 'semi-rigid,' is passed through a series of saws which cut the material to the required dimensions, following which it is packed by hand into cartons. This is the first time the material is handled in the process. The desired bulk-weight is obtained by relating the conveyor speed rate to the output of the cupola, taking the desired thickness into account.

The semi-rigid type has not sufficient inherent strength to be used without support and to meet this need a moulded form of insulation is made. This material is made in two grades—that necessary to withstand high temperatures up to 1,500°F. and that designed for cold work. The process is the same in each case, the only difference being in the binder material used.

Moulding process. In this process a suspending agent is added to a large tank of water and the mineral fibre is then added. It is dispersed under agitation and maintained in suspension. Specially formulated adhesive, rendered water-soluble, is added and thoroughly dissolved in the water. When dispersion is complete, the adhesive is precipitated by chemical action, whence it coalesces with the fibre. This attachment of adhesive to fibre is complete, for these two carry opposite electrical charges which helps to give complete combination. After giving the charge a few minutes to ensure the precipitation is 100%, it is run into perforated moulds which allow a certain amount of moisture to drain away. The remaining moisture is dried off and the adhesive is baked up in ovens.

The moulds are designed to give lags for all standard size pipes, in half-sections, quadrants, hexagonal, octagonal, or an even greater number of sections, if the pipe is very large. The moulds are also made to give blocks of 6 by 24 in. up to 24 by 36 in. at 6-in. intervals, from $\frac{1}{2}$ to 2 in. thick.

Another series of products is that wherein the fibre is contained in covers. The type of cover used is dependent on the duty for which the insulation is required. Examples of the covering materials are:

(*a*) Bituminised paper.
(*b*) Flame-proofed scrim cloth.
(*c*) Asbestos cloth.

All used for medium-temperature insulation.

At temperatures above the limit which these materials will bear, the mineral wool is contained in chicken-wire mesh.

The first three of these products are made on the conveyor-belt system. The bottom cover is laid down, the wool rolled on to the cover, and finally the top cover put on. All this takes place as the material is moving forward to an industrial sewing machine which stitches up the insulation. The material can be made totally enclosed or with open edges, as desired by the customer. It is available in standard sizes and, at a slight extra cost, in larger sizes.

The material, made wire-mesh-enclosed, is ideally suitable for rough service conditions, and it has a limiting temperature of 1,500°F.

In this case, stitching has to be carried out by hand, as the needles of an industrial sewing machine are liable to be damaged by the wire mesh. The sewing is carried out using a lacing wire between top and bottom layer of the wire mesh.

Flexible felted sections are made for pipe insulation, and another form for pipe covering—strip insulation, sewn in flame-proofed scrim cloth—is manufactured. This is made 4 in. wide and 18 ft. long. It is applied by winding helically round pipes.

In addition to the industrial products, a cylinder jacket is manufactured which is used for the insulation of the domestic hot-water cylinder. This jacket is finished

in leathercloth and enhances the appearance of that particular part of the house.

To complete the range of products available to industry, an **insulating cement** is manufactured. This product is used both as an insulant on bare surfaces and as a finishing coat on other insulating materials.

Manufacture is carried out in large rotary drums. Proportions of pelleted mineral wool, followed by a mineral filler and a mineral adhesive—a volcanic clay—are added. These are thoroughly mixed in the drums, then tipped into bags. All that is required before application of this material is that it be mixed with the amount of water indicated on the bag. When dry, this material sets very hard. It can be coated with a protective coating of bitumen or paint, as so desired.

Applications

It cannot be too strongly emphasised that extreme care should be taken in fitting the insulation material.

All operating equipment should be tested for leaks and mechanical defects before applying the insulation. Above all, the surface should be clean. The application of the insulation and accessories should be carried out in a neat and workmanlike manner.

Adequate means should be provided to maintain the insulation in contact with the surface, in order to prevent accidental displacement. It is strongly recommended that the insulation should be fully enclosed by protective layers of bituminous or cement compositions or, most permanent of all, sheet metal cladding.

On vertical surfaces, blanket or mattress insulation, semi-rigid and moulded insulation may be supported by horizontal angles, studs inserted through the insulation by a welding gun, or by wires held perpendicular to the surface on which the insulation can be impaled.

On horizontal surfaces having insulation on the under-side, the attachment devices should be closer together to prevent sagging. In addition, the attachments should carry washers to spread the weight of the insulation more evenly. On horizontal surfaces with insulation on the upper side, fastenings are only required against lateral displacements, due to accidental contact, and to guard against vibration wandering. If possible, circumferential fastening by bands or wire should be installed *in addition* over the insulation to ensure permanent contact.

On rectangular equipment, such as ducts or breechings, it is recommended that corner beading should be installed.

Only if the installation is properly carried out will the advantage of very low heat losses consistent with the quality of the mineral wool insulation be apparent.

Mineral wool in its various forms is used over a very wide field in industry. It is used in a bed-warmer which is so made that it gives out a low heat over a long period, and it is used in the insulation of very large-scale plant such as chemical works, oil refineries, shipping and cold stores. It finds application in nearly all constructional work where insulation is required, and in mass-production equipment, refrigerators, gas, electric and solid-fuel cookers.

Table 1. Recommended Densities for Mineral Wool Insulating Materials

		Temperature °F.				
		Below zero to 300	300 to 350	350 to 550	550 to 1,000	1,000 to 1,500
Density Lb./cu.ft.	Wool	10—12	10—12	12—14	14—16	16—18
Semi-rigid		6—9	10—12	12—13	14—16	16—18

Table 2. Recommended Thicknesses for Mineral Wool Insulating Materials except Cement

Pipe bore inches	*Internal temperature range* °F.						
	70–200	200–400	400–600	600–800	800–1,000	1,000–1,200	1,200–1,500
Up to ¾ ..	½	¾	¾	1	1	1½	2
1—2 ..	¾	1	1½	1½	2	2	2½
2½—3½ ..	1	1½	1½	2	2½	2½	3
4—6 ..	1	1½	2	2½	3	3	3½
7—12 ..	1½	1½	2	2½	3	3½	4
Vessels & flat surfaces ..	1½	1½	2½	3	3½	4	4½
Efficiency (%)	85—90	91—96	96—98	97—98	97—98	97—98	97—98

Table 3. Recommended Thicknesses for Mineral Wool Insulating Cement

Pipe bore inches	*Internal temperature* °F.							
	100	200	400	600	800	1,000	1,200	1,400
Up to 2	½	¾	1	1¼	1¾	2	2	2
2½—4½	¾	1	1½	1¾	2½	2½	3	3
5—9	1	1	2	2½	3	3	3½	4
Over 9	1	1½	2	2¾	3½	3½	4	4
Vessels & flat surfaces	1	1½	2	3	4	4	4½	5
Efficiency (%) ..	85—90	87—92	90—94	92—95	94—97	96—98	97—98	97—98

Besides its use in thermal correction work, it is widely applied for acoustic correction and sound deadening in film and radio studios, rolling stock and road vehicles and, at the present time, it is being applied to the concert hall under construction for the 1951 Festival of Britain. The absorption coefficient over the audible frequency range is between 80 and 95%, depending on the sound frequency.

Filtering

Another very wide application is as a medium for filtering gases and non-aqueous liquids. The large-scale production of *Perspex* by a continuous process was rendered possible by the application of mineral wool as a filter medium to remove dust particles from the atmosphere; otherwise *Perspex* would have been contaminated.

In the manufacture of penicillin, the mineral wool, after modification to suit the application, was found entirely satisfactory for removing all bacteria and spores which would upset the growth of the sensitive penicillin mould. Sterile air can be obtained by filtration through this material with an efficiency of between 99.998 and 100%.* Corrosive gases up to 1,000°C. can be filtered safely.

These examples are given to illustrate the versatility of this product, but the main output is absorbed in thermal correction.

Thermal correction

Mineral wool, judiciously applied, can save in capital expenditure by reducing the size of plant necessary to give a stated output. In addition, having a very low thermal conductivity, it does not require a great thickness to give protection against heat losses and, with its exceptionally high limiting temperature (1,500°F.) the thickness of refractory brickwork can be reduced, thereby saving further on heat losses normally required to heat up the brickwork.

Bearing these facts in mind, it can be readily understood why it finds use in chemical plant, locomotives, annealing furnaces, boilers, power stations and a host of other high-temperature installations.

Its ease of application also renders it specially suitable for motor vehicles, aircraft, railway carriages, containers and buildings, for all insulation of plane surfaces, curved surfaces, pipes and ducts.

*'The Removal of Micro-Organisms from the Air by Filtration,' by S. G. Terjesen and G. B. Cherry. Paper read before the N.W. Branch of the Institution of Chemical Engineers, Oct. 11, 1947.

Chemical Engineering at Low Temperatures

By N. W. Roberts, M.A., F.R.I.C.

CHEMICAL reactions are rarely, if ever, carried out below -50°C. Yet a large and growing number of 'chemical plants' are in operation all over the world at temperatures which may be as low as -200°C. Such plants are engaged on the physical separation of commercially valuable constituents from gas mixtures. The most important of such gas mixtures is air, from which oxygen, nitrogen, argon, neon, helium, krypton and xenon are extracted. Second in economic importance is coke-oven gas, from which hydrogen, ethylene and methane are commercially extracted. The separation of gases derived from petroleum has traditionally been carried out near or above room temperature, using high pressures to liquefy the gases to be treated.

Low-temperature plant presents a different appearance from plant performing the same unit operation at ordinary temperatures. This fact, coupled with the fact that low-temperature plant only effects physical changes, led to the suggestion about four years ago that the name 'physical engineering' should be given to this branch of science. The distinction did not become popular, because the principles on which low-temperature plants are designed are identical with those used in the design of ordinary chemical plant, differences in detail being occasioned by the low temperature at which the plants work.

Like any other plants, low-temperature plants are designed on the basis of an optimum balance between capital and operating costs. The most important item in the operating costs is power. That this should be so can be readily demonstrated if we remember that, in order to cool gases down to low temperatures, we must have refrigeration, to obtain which work must be done; and that, in order to perform a physical separation without contravening the second law of thermodynamics, further work must be done. Since the only way in which work can usefully be done on a gas is by compression, it follows that the power supplied to the compressors of the plant has a great influence on the ultimate cost of the separation.

Refrigeration

Considering first the problem of refrigeration, it is well known that the theoretical work W required to produce a quantity of refrigeration Q at an absolute temperature T_2, operating with a heat sink at an absolute temperature T_1, is given by

$$W/Q_2 = (T_1 - T_2)/T_2$$

The effect of this relationship on the power consumption of refrigerating machines is shown in Table 1, which gives the relative theoretical power consumptions to produce equal amounts of cold at various temperatures. The heat sink is taken as 27°C., and the power consumed in producing cold at -10°C. is taken as unity.

Chemical engineering design of plants working below –50°C. is based on the same principles as apply in other fields, but in practice the plants appear very different. These differences are neither fads of the designers nor slavish adherence to custom; they are founded on firm physical principles. In this article some of the variations in procedure are outlined and explained.

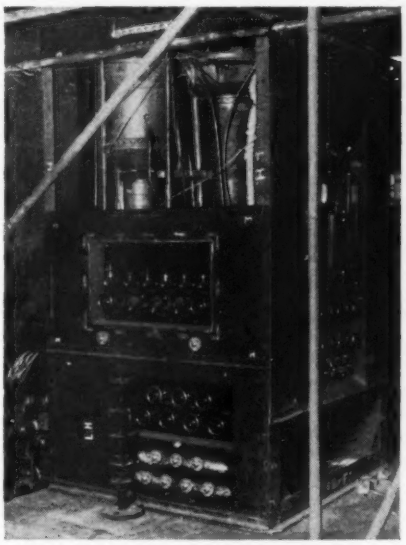


Fig. 1. Air separation plant in course of construction.

TABLE 1

T°C.	T°K.	*Power*
—10	263	1.00
—50	223	2.49
—100	173	5.29
—150	123	10.37
—200	73	22.37

The rapid increase of the relative theoretical power consumption as the temperature falls shows the importance, when working at low temperatures, of thermal insulation and, indeed, of reducing losses of cold from all sources to a minimum. In practice, refrigeration efficiency tends to be lower at lower temperatures, so that the trend is accentuated further.

For this reason it is usual for low-temperature plants to be built within the smallest space possible, without more than a passing regard for accessibility and ease of maintenance. All the parts working at low temperatures can then be surrounded with a steel box which is packed with insulating material, which may on occasion be more than 1 ft. thick.[1] (This is clearly shown in Fig. 1, which shows an air-separation plant in course of construction.)

Heat exchangers

The high power consumption of cold production also affects the design of heat exchangers, for it will be clear that imperfect cold recovery due to the temperature differences at the warm ends of the heat exchangers results in a loss of cold just as real as that due to imperfect thermal insulation. For this reason it is common to find exchangers designed to give warm-end temperature differences of 1°C. or lower, and very rare indeed to find warm-end temperature differences of more than 5°C.

Heat exchangers for low-temperature work are very different in appearance from those used at ordinary temperatures. In designing a heat exchanger for ordinary temperatures, the chemical engineer has to pay attention first and foremost to the need for easy cleaning; hence the continuing popularity of the shell and tube exchanger. The low-temperature engineer also has to bear in mind the possibility of fouling of his heat-transfer surfaces. At low temperatures, however, the important fouling agents are substances like water and carbon dioxide which condense to the solid state and, if not dealt with, not only foul the surfaces but block the tubes as well. The low-temperature engineer has, however, the advantage that these fouling agents can be easily removed by warming the apparatus to ordinary temperature, during which process the offending substances melt or vaporise and can thus be readily dealt with. In consequence, the only anti-fouling provision which is necessary is the furnishing of facilities for periodical thawing of the unit. During the thawing process the unit is, of course, off stream, and its necessity therefore constitutes a serious charge on the process. Careful attention to the details of the purification process has, however, been markedly successful in increasing the length of run between thaws; in one case, a unit was kept on stream for twelve months.

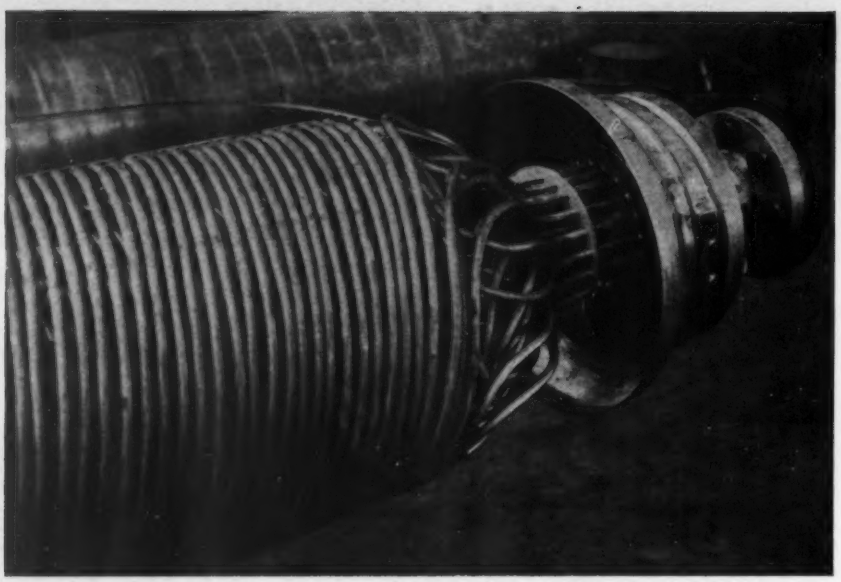


Fig. 2. Typical heat exchanger for low-temperature work under construction.

Heat exchangers for low-temperature work are of three broad types, high-pressure exchangers, regenerators and reversing exchangers. The latter two have the advantage of being self-cleaning. This means that the air supplied to these exchangers need not be purified; it deposits its water vapour and carbon dioxide on the surfaces of the exchangers, whence they are later revaporised into the product stream by an ingenious control of heat-exchange conditions. Such exchangers have been fully described in the literature.[2, 3]

The type of exchanger most frequently used for the exchange of heat between a high- and a low-pressure gas is due to Giauque. The author is not aware of its use apart from the low-temperature field, almost certainly because once fouled by non-volatile materials it is quite impossible to clean. The arrangement is occasioned by the low specific volume of compressed gases, which makes it difficult to obtain in conventional arrangements the high velocity within the tubes which is essential for efficient heat exchange without incurring prohibitive pressure drops on the low-pressure side; readers will remember that Nature has somewhat unfairly provided that the lower the pressure the higher the pressure drop for a given mass flow of gas in a given arrangement. In order to overcome this difficulty, the high-pressure tubes are spirally wound round a mandrel, and the low-pressure gas is led across the turns in a direction parallel to the axis of the mandrel, so that at all times the flow of gas is nearly perpendicular to the tubes. In order to avoid spiralling of the low-pressure gas, alternate layers of high-pressure tubes are wound in opposite senses. By variation of the number and diameter of tubes, number of layers and of the radial and axial pitch of the tubes it is possible to tailor exchangers for a given duty. A typical exchanger of this type is shown in course of construction in Fig. 2.

Constructional materials

Materials of construction for low-temperature work are limited by the lack of impact strength of many metals, notably mild steel, at low temperatures. Among the materials which are satisfactory in this respect are copper, aluminium, stainless and some other alloy steels. Copper has been the traditional material of construction in this industry for many years, and has many advantages to commend it. It can be soft soldered, a method of construction which is convenient particularly for the joining of parts of a metal of exceptionally high heat conduction; its mechanical properties at low temperature are satisfactory; and it is resistant to most of the corrosive influences met with in the industry. There are signs, however, of a questioning of the predominance of copper; aluminium is freely used in the large new American plants, for instance.

Distillation

Distillation processes at low temperatures are subject to the same laws as those at ordinary temperatures, but the plants in which they are carried out show substantial differences in arrangements and in detailed construction. The differences in arrangement are occasioned by the problem of reflux. At ordinary temperatures this problem arises only in a minor degree. The reflux ratio is decided on the basis of a balance between capital and operating costs—smaller reflux, smaller steam and cooling water costs, but a longer column. At low temperatures the same balance is made, but other considerations enter into the account. At low temperatures the provision of reflux is no longer a matter of steam and cooling water; condensable gases must be used, which have to be compressed into the apparatus and cooled to the working temperature before they can be admitted to the tubes. The provision of reflux, involving as it does compressors and heat exchangers, is therefore an expensive matter.

Fortunately, the boiling points of the constituents of air are fairly widely spaced; their relative volatilities are, in fact, usually high enough to allow separation at such low reflux ratios that the latent heat of the

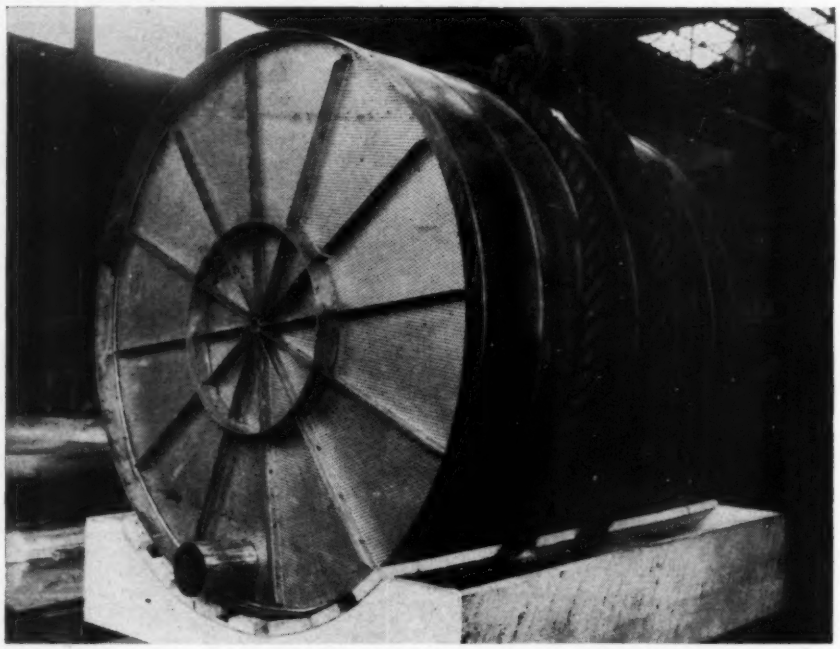


Fig. 3. Underside of perforated plate of distillation column for low-temperature processes.

gas to be separated is sufficient to provide the reflux for the separation.[4] Sometimes, indeed, it is more than sufficient, in which case part of the gas can be expanded in an engine or turbine, so providing refrigeration. This is not the place for an account of the many ingenious devices which have been used to squeeze the last erg of useful work from the compressed gas; those interested can find further information in the references cited.[4, 5, 6]

Absence of bubble plates

Distillation columns for low temperature seldom contain bubble plates, though this is partly due to the fact that cleaning is seldom required, for reasons outlined above; the main reason is the necessity to reduce to the last degree the column height and hence the loss of cold from the column to the surroundings. In order to achieve such a reduction of height, it is essential that the trays used shall be of the highest efficiency, and in order to secure this the liquid on the trays is given a directed flow.

In the theoretical plate, in terms of which most distillation calculations are carried out, the vapour bubbles through the liquid, which is assumed to be perfectly mixed, and emerges in equilibrium with it; since the liquid is perfectly mixed the vapour therefore emerges from the plate in equilibrium with the liquid leaving. In the plate with directed flow, the vapour bubbles through the liquid and emerges (theoretically) in equilibrium with the liquid at the point of contact. Since the liquid contains, on the average, more of the more volatile component than the liquid leaving the plate, it follows that the effect of the plate is greater than that of a theoretical plate.

It also follows, however, that since the liquid has a longer path on the plate, its hydraulic gradient will be greater; and since, in order to save height, the plate spacing is reduced as far as possible, the hydraulic resistance of the plate assumes an importance greater than that in normal columns, if flooding is to be avoided. For these and other reasons, the perforated plate is used in preference to the bubble-cap. Fig. 3 shows a view of the underside of a perforated plate.

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'I.C.E.' February

Besides our usual features, there will be a special section devoted to **DRYING**

The East German Chemical Industry

THE East German five-year plan, to start this month, aims at almost doubling chemical production. The 1950 target figures for a number of important chemicals, compared with the 1955 target figures, were set as follows (in tons):

	1950	1955
Sulphuric acid ..	256,000	400,000
Sodium carbonate	102,000	380,000
Caustic soda ..	147,000	250,000
Potash	Unknown	2,000,000
Nitrogenous fertilisers	208,000	235,000
Phosphatic fertilisers	48,000	93,000
Soap	36,000	100,000
Petrol (synthetic)	446,000	780,000
Synthetic rubber	38,000	60,000

In order to achieve the increase in production, rebuilding of existing plants and the building of new ones is scheduled. In addition, the development of new methods for producing heat-resisting varnishes, lubricating oils and chlorine chemicals, and research into the industrial synthesis of fatty acids, antibiotics, anti-tuberculosis, anti-cancer and other drugs will be undertaken.

Raw material shortages

It is generally believed that the shortage of sulphuric acid has been overcome, according to the West German economic monthly, *Wirtschaftsdienst*. Gypsum, which is obtainable in sufficient quantities, has replaced rare pyrites as raw material. Three furnaces with a capacity of 24 tons each, were built in 1949 in the sulphuric acid plant at Salzwedel.

The East German chemical industry cannot produce several organic chemicals, because certain raw materials are unobtainable in sufficient quantities from East Europe or at all from the West. The industry has no borax, no shellac and no gum arabic. The lack of hard coal prevents it from producing organic dyestuffs and numerous pharmaceuticals. Synthetic fibres can be produced only on the basis of phenolic resins, due to the lack of urea. The glass and detergents industries have insufficient quantities of sodium carbonate, and there is little caustic soda for the production of synthetic fibres and for the soap industry.

Production of sodium products will be doubled if the plan succeeds. Rock salt and lime, of which there are adequate deposits in East Germany, are now being used in soda production.

The East German chemical industry is organised in three sectors: State-owned Soviet companies, 'people-owned' plants and private industry.

Soviet-owned plants

The Soviet companies include the East German decartelised plants of the I.G. Farben combine, such as the petrol and fertiliser plant at Leuna, the synthetic rubber plant at Schkopau, the electrochemical combine at Bitterfeld, the Agfa and dyestuffs factories, both at Wolfen, and the nitrogen works at Piesteritz. The Soviet companies represent about 35% of the whole chemical industry.

The most important of them produce: **Leuna:** petrol, nitrogenous and phosphatic fertilisers, phenols, cresols, basic materials for synthetic fibres, amines, acids and alkalis; **Schkopau:** synthetic rubber, *Igelit*, the synthetic fibre *PCU*, formaldehyde, lubricating oils, carbides, pharmaceuticals, organic and inorganic products; **Bitterfeld:** inorganic chemicals, including iron derivatives, potash, other potassium salts, compressed gases, light metal alloys; **Agfa:** films, photographic chemicals and synthetic fibres; **Wolfen:** cosmetics, pharmaceuticals, dyestuffs, varnishes and tannery products.

'Peoples' plants

The 'people-owned' chemical plants are controlled by the chemical department of the East German Ministry of Industry. This industrial sector is divided into ten central associations of 'people-owned' factories (Vereinigung Volkseigener Betriebe-Zentral or VVB-Z) with a total of 162 plants and about 33,800 employees. Similar associations, controlled by the respective regional governments (VVB-Land) have been set up in each of the six states in East Germany. The six VVB-L represent 167 smaller plants with about 15,000 employees. The ten centrally controlled VVB-Z are: VVB 'Alcid,' for organic chemicals, with 14 factories and 3,520 employees; VVB potassium and salts, with 17 factories and 9,340 employees; VVB dyestuffs and varnishes, with 28 factories and 2,330 employees; VVB coal and carbon products, with 11 factories and 4,860 employees; VVB 'Organa,' with 7 factories and 3,700 employees, producing organic and pharmaceutical chemicals; VVB 'Oxyka,' with 14 factories and 570 employees, producing compressed gases, oxygen, nitrogen, hydrogen and carbides; VVB 'Pharma,' with 11 factories and 11,510 employees, producing pharmaceutical chemicals, vaccines, cosmetics and drugs; VVB 'Plasta,' with 10 factories and 950 employees, producing synthetic fibres and artificial resins; VVB 'Sapotex,' with 14 factories and 4,710 employees, producing soaps; VVB 'Variochem,' with 25 factories and 2,310 employees, producing miscellaneous chemicals.

About 30% of the chemical industry is privately owned, consisting of minor factories, and is not included in the five-year plan. This sector of the chemical industry faces difficulties in obtaining raw materials and bank loans. The East German chemical industry as a whole employs about 150,000 people.

SIZE REDUCTION

Jet mills, homogenisers, particle size measurement, explosion and fire risks

By R. V. Riley, Ph.D., B.Sc., A.I.M.

A PREVIOUS review[1] contained a reference to a text book, 'Handbook on Mineral Dressing,' by A. F. Taggart, published in 1945. During the past year an important chemical book of reference has been extensively revised and is issued as a third edition, namely Perry's 'Chemical Engineers' Handbook,'[2] which now contains a chapter on size reduction and size enlargement. To this reviewer the concept 'size enlargement' appears novel; the antithesis of size reduction, it covers many operations of interest to the chemical engineer such as compacting, flocculation, nodulising, sintering, casting, precipitation, etc.

The section on size reduction contains ten separate papers, each written by a specialist, and some 300 references are quoted. These informative articles cover 70 pages of the handbook, which itself contains 1,942 pages of closely set type. Size reduction is classified under the headings:

Primary and secondary crushing.
Grinding (pulverisation and disintegration).
Cutting.
Machining.
Flaking.
Emulsification.
Spraying.
Gas dispersion.

It is not, however, the purpose of the present reviewer to adopt this classification. The wider aspects recognised by Perry's handbook are too extensive and too specialised to treat adequately here.

Perry gives a classification of crushing and grinding equipment, together with a description of the uses of each unit. Here it is:

CLASSIFICATION OF CRUSHING AND GRINDING EQUIPMENT

Crushers

(*a*) JAW. Blake and Dodge are common types. Blake for high production and non-clogging; Dodge for low production, intermittent service and higher reduction ratio.

(*b*) GYRATORY. Replacing jaws as primary crushers. Three general types are suspended, supported and fixed-spindle.

(*c*) CONE. This is a type of gyratory crusher. Successor to disc crusher.

(*d*) PAN. Edge runner and chaser mills are variations. Can be operated wet or dry.

(*e*) ROLL. (1) *Smooth.* (2) *Toothed.* Smooth rolls for hard and soft materials; toothed rolls for soft materials. Toothed rolls available in single double and multiple arrangement.

(*f*) ROTARY. Suitable for coarse crushing of soft materials.

(*g*) HAMMER. Heavy construction with breaker plates, sturdy hammers, bar grate discharge. Operates at low speeds.

Grinders, mills or grinding mills

(*h*) BALL. (1) *Ball.* (2) *Pebble.* (3) *Rod.* (4) *Tube.* (5) *Compartment.* Length equal to diameter with large balls for crushing. Length equal to diameter for batch grinding. Pebble mills used to avoid iron contamination. Length, two to three times diameter for open- or closed-circuit operation. Rod mill produces minimum 'fines'; can operate with moist material; rods instead of balls; length is twice diameter. Ball, pebble and rod mills operate wet or dry. Tube and compartment mills have length greater than twice the diameter. Compartment mill is tube mill divided into zones with decreasing size of balls from feed end. Frequently operated in closed circuit with size classifier.

(*i*) RING-ROLLER. (1) *Vertical ring.* (2) *Horizontal ring.* (i) Bowl. (ii) Ball and ring. Generally incorporate air or screen classification. Adapted to drying while grinding. Either the ring may rotate, or the rollers revolve. Spring pressure or centrifugal force may hold rollers against ring.

Crushers

(*j*) HAMMER. (1) *Rigid hammer.* (2) *Swing hammer.* (3) *Ring hammer.* (4) *Multiple-cage.* (5) *Disc (toothed plates).* Also called impact mills. Smaller clearance between grate bars than for crushing and higher speeds. Often have perforated plate or screen discharge. Called beater mills with rigid hammers or beaters. May have horizontal or vertical shaft. Multiple-cage disintegrator and toothed-plate disc mill are really hammer or impact mills.

(*k*) DISC (ESSENTIALLY SMOOTH PLATES). (1) *Single runner.* (2) *Double runner.* (3) *Buhrstone.* Double runner gives higher relative speeds. Also known as attrition mills or plate mills. Buhrstone mill is in this category. Can be operated wet or dry. Colloid mills may be included in this class.

(*l*) FLUID ENERGY (JET MILLS). (1) *Gas.* (*i*) Air. (*ii*) Steam. No moving parts in mill. Heat-sensitive materials can be pulverised with cooled air. Steam is superheated to avoid condensation on product.

An interesting, though less comprehensive, book published at the end of 1949 is the monograph 'Industrial Grinding and Reduction Plant,' by C. S. Darling.[3] Its author describes in fair detail the types of mills available for crushing minerals, especially coal, namely ball mills, ring roll mills, rotary impact mills and finally fluid jet mills. The treatment of the latter type of grinding mill is of particular interest as it is one of the few adequate treatises dealing with recent developments in this field.

Fluid jet mills

Details are given in Darling's book of the *Micronizer*, *Kidwell* mill and *Blaw-Knox* mill. These mills contain no mechanical moving parts. The material being crushed is steam- or air-borne and passes spirally through a flat ring-like chamber as in the *Micronizer* or around a closed-loop circuit of pipes, as in the *Kidwell* mill, or is circulated by a combination of the two systems as in the *Blaw-Knox* mill.

Jet pulverisers open up new and exciting possibilities. A coal atomiser for the pulverising of crushed fuel and its simultaneous combustion in a gas turbine is an example of one current line of thought in America. The development consists of a venturi-shaped nozzle through which passes a stream of high-pressure air carrying crushed coal. A pressure drop of at least 80 lb./sq.in. occurs at the venturi throat and this results in a shattering effect upon the solid particles, due to internal expansion. It is expected that this type of atomiser will deal with 1 to 1.2 lb. of coal per lb. of air and supply the fuel/air mixture direct into the combustion chamber of the turbine.

Electrostatic charging of small particles occurring naturally in these fluid jet mills may be employed for classifying large and small grades of crushed material. If the mills are constructed of iron or other metal the finer particles cling to the sides

TABLE 1. 'MICRONISER' PERFORMANCE*

Material	Product average size	Feed		Fluid consumption lb. fluid/lb. solid	
		Size sieve No.	Rate lb./hr.	Air	Steam
Ceylon graphite	2	3	200	—	8.5
Cryolite	3	60	900	—	4.0
Limestone	3.5	80	1,000	—	4.0
Hard talc	3.5	20	1,000	—	4.0
Silica gel	5.5	8	500	—	3.5
Soft talc	6.5	20	1,800	—	2.5
Barytes	3.5	40	1,800	—	2.2
Bituminous coal	2	10	1,300	—	1.2
Copal resin	5	2	600	7.5	
Wolframite ore	5.5	10	800	5.6	
Sulphur	3.5	3	1,300	3.5	

Ind. Eng. Chem., 1946, **38**, p. 672.

of the plant and are held there by electrostatic charges; alternatively, if the mills are made electrically non-conducting by lining with rubber or ebonite the inner surfaces remain clean and uncontaminated with fines. This phenomenon may be used either to separate graded particles or to prevent the clogging of the mill casing or distributing pipes.

C. E. Berry, writing in Perry's handbook, also gives an interesting description of fluid energy and jet mills, and a table reproduced on the opposite page gives interesting data upon the performance of the *Microniser* working on materials of $\frac{1}{4}$ in. average particle size.

Another type of jet-mill is the subject of a British patent by H. A. Procter,[4] in which it is described as follows:

'Frangible minerals are reduced to a state of division in the sub-sieve range by treatment in a non-rotary apparatus with pressure fluid (air, super-heated steam) so that the flow of the material with the pressure fluid is spirally around the centre line. The material passes from the pulverising chamber to a separate classifying chamber and insufficiently reduced material is returned to the pulverising chamber. In the pulverising chamber, the travel path of the material continuously varies by an arrangement of deflecting ledges or ribs in a spiral tubular channel, rising vertically to a larger diameter of the windings into which spirally arranged jets discharge the pressure fluid.'

A mill of similar type but grinding down to 40μ only is described by C. H. Wheeler.[5] The chief advantage of this mill is its lower power requirements.

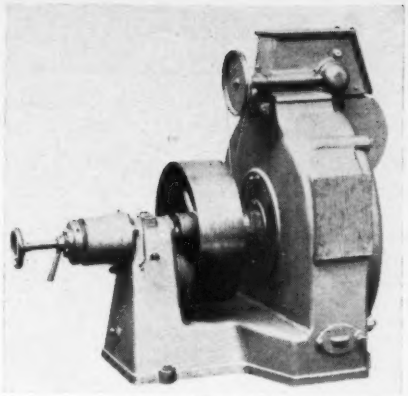
The Wheeler mill and the *Microniser* have been applied to the grinding of talc to $1\frac{1}{2}$ to 20μ size.[6]

Homogenisers and colloid mills

The preparation of an emulsion of two or more immiscible liquids is in many ways a similar operation to the fine grinding of a solid in a fluid energy mill. The initial disintegration of the liquid is accomplished more easily, but to maintain a stabilised emulsion is more difficult than to keep a free flowing powder. Surface-active agents are widely used in emulsion preparation.[2] Most conventional types of homogeniser employ methods of hydraulic shear to bring the dispersed phase into a state of fine sub-division of the order of 1μ. This shearing action in the liquid is brought about mechanically by interference clearances between moving and fixed parts of the mill, by pressure release or by fluid impact.

Theory of ball mills

The theory of the ball mill has been re-studied by two Russians, Levenson[7] and Neronov,[8] working separately. Cinematograph photographs were taken to record ball movement in cylindrical mills. The movement of the balls was resolved into three stages: (*a*) along the wall of the drum; (*b*) away from the wall of the drum and along a parabola; and (*c*) along the catenary. Levenson's theory claims 100% agreement with practice, although there appears to be some grounds for criticism of the theoretical processes of both workers.



[Photo: Thomas Robinson & Sons Ltd.
Universal cereals grinding machine.

An article by Klovers[9] describes an interesting practical method, using log-log graph paper, of predicting the range of sizes to be expected from any crushing and grinding mill given the size of the feed. This method presumes a knowledge of the performance of the mill on similar grist and of a size not too far removed from that of the desired product.

Vibratory ball mill

The German war-time researches into the vibratory ball mills, to which reference was made in an earlier review,[1] have encouraged at least one British manufacturer to incorporate these principles in a new laboratory ball mill.[10] In this mill the pot is suspended horizontally on springs and by means of an out-of-balance weight, the axis of the mill sweeps out a cylindrical path, causing 'gravity assisted' projection of the balls with increased efficiency and rapidity of grinding. This mill has been successfully applied to such materials as tungsten ores, rutile, ilmenite, coal, coke, alumina, boiler scale, slags, etc. The closed pot makes it of value in moisture determination where other grinding appliances may be unsuitable.

Clay shredders

Producers of raw materials are becoming more aware of the need for 'window dressing,' formerly only necessary for the sale of consumer goods. Kaolin clay as a filler for rubber is now supplied by American southern states producers,[11] dry-milled in 50-lb. bags ready for immediate use. Formerly the clay was shipped crude from the mine. First stage in the preparation of the finely ground clay by fully mechanised methods is a 'slicer' of new design.

The 'slicer' is similar to a pan mill with revolving bottom plate but no runners. Numerous knives protrude upwards from rectangular slots like the corrugations in the household suet shredder. When raw clay is dumped upon this revolving disc it is shaved by the knives, passes through the slots and is swept into the bottom discharge opening by rotating wiper arms. The size of the product can be altered by adjusting the height of the knives above the plate in the same way as the carpenter is able to vary the depth of the cut with an ordinary wood plane.

The size and design of the 'slicer' varies. A 4-ft. diameter machine rotates at 50 r.p.m., driven by a 50-h.p. motor, and should handle 20 to 30 tons/hr. of clay. There may be as few as 32 knives or as many as 64 arranged radially or along two or four chords of the circle. For some clays, baffles are used to guide the clay on to the knives and to prevent balls from simply rolling round the mill. In other cases these are unnecessary. This type of mill will deal with clay over a wide range of moisture content.

Another new design of clay shredder, working on similar lines, powered by a 30-kW electric motor and producing 15 to 20 tons/hr. of powdered clay, has been described by Malchenko.[12]

The importance of sizing of solids which are to be employed in chemical reactions in fluidised beds is now well recognised. To secure the necessary fineness and uniformity, modern crushing and grinding equipment is essential. Carefully sized metal powders and refractory materials are in demand for the new 'Ceromets' developed for use in gas turbines and for other special applications where refractories of high thermal conductivity and strength are required.[13]

A new concept of hardness scale

Dmitriev[14] has proposed a new hardness scale for mineralogical materials to replace the old Mohs scratch hardness scale. The hardness of all materials is expressed in terms of the resistance to penetration by a diamond pyramid as kg./sq.mm.:

$$\text{Micro hardness} = \frac{\text{Sin } \alpha \, P}{d^2}$$

where α = included angle between opposite facets of the pyramid, usually 136°; P = load in kg.; and d = diagonal of impression.

Some common minerals tested by Dmitriev and also by Prof. Krushchov, using a similar technique, gave the values for hardness recorded in Table 2 overleaf.

This indentation hardness test has obvious advantages, as the numerical hardness readings are on the same scale as is adopted for metals. A hardened steel or iron, for example, would give a reading in the range 500 to 1,000.

Particle size measurement

The fineness of grinding of paints, enamels and printers' inks may be determined by measurements made on a modified roller-type Hegeman gauge

block.[16] The gauge consists of a steel block, upon the flat surface of which is inscribed straight ribbon-like channels 1 in. wide and up to 10 in. or more in length. The depth of the channels ranges from 0.001 in. at the deepest end to zero at the shallow end. The gauge block is graduated over the length of the channel into ten divisions. Each division therefore represents a change in depth of 2.5μ.

The method of using the modified gauge consists in applying by means of a roller a layer of paint or similar media under test and then following the applicator roll by a plane knife blade which scrapes off the surplus material. The maximum size of the pigment particles or aggregates may be estimated by the length of ribbon remaining on the gauge block. A more exact estimate of the particle size may be obtained by noting the onset of scratching of the remaining pigment film. This may be viewed conveniently under the microscope.

Bernstein[15] discusses in detail the theory underlying this test, showing the importance of particle shape upon the onset of scratching of the pigment film during the test. He also demonstrates that the test measures the aggregate size rather than the true particle size and that the results depend upon the degree of dispersion in the media. It is demonstrated that with very fine suspensions the particles may form aggregates or 'impactoids,' which are sometimes surprisingly large.

The test may be used not only for estimating the fineness of grinding of paints, inks, etc., but also for evaluating the fineness of dry milled powders which, for the purpose of the test, are dispersed in a liquid. It is appreciated that this gauge method, at least for printing ink pigments, has the limitation that it evaluated only the oversize range as a criterion of fineness, rather than the entire particle range size.

Particle size determination in paints and pigments is, of course, attended by difficulties, due to the agglomeration of ultimate particles. Some form of dispersion agent is usually necessary before the standard methods can be used.[16]

For the rapid assessment of particle size in dry powders the microscope is most suitable. In one recent method used for clays, the powder is compared directly with a Cylycopodium powder of known size.[17] The distribution of dust particles in air has been determined by scanning the cross-sectional area of a vessel by an electron beam from a cathode ray tube and measuring the response upon a photoelectric cell which is connected to an electronic counting circuit.[18] Sizing by sieve analysis was recently studied scientifically by Mörtsell.[19] He concluded that there was a critical loading factor for test sieves and, provided this was taken into account, hand sieving could be standardised so as to give results as accurate as were obtainable by machine sieving. A new graphical method of interpreting Stokes' formula for the settling of suspensions within the particle size limits of 50 to 5μ has been described by Martens.[20]

Fine grinding of ironstone

In America just now the problem of how to use the abundant supplies of taconite iron ore to replace the dwindling reserves of present best-quality Lake Superior ores is under consideration.[21] Contrary to the present blast furnace practice of utilising ore in lump form, it will be necessary to crush these ores down to particles of 20μ or, better still, 5μ, to free the magnetite from the silica gauge. At present it is planned to effect a 90% iron recovery and to produce a concentrate containing 64.5% Fe by crushing to −325 mesh. This reduction is carried out in four stages by:

(1) 60-in. Gyratory crusher.
(2) 24-in. Reduction crushers.
(3) 7-in. Symons short-head cone crusher.
(4) Rod mills and ball mills.

The ore is crushed dry in the first three stages; in the rod and ball mills, however, it must be dealt with wet to reduce dust.

The rod mill product has an average size of −6 mesh and, after magnetic separation, the concentrate is further ground in the ball mills in closed cycle with a classifier to the required fineness.

Another interesting paper dealing with the more conventional crushing of ironstone to lumps 1½ to 2½ in. was published recently by the British Iron and Steel Research Association.[22] The aim of the investigations was the avoidance of fines. It was concluded that a combination of primary jaw and secondary cone crushers were most satisfactory from this point of view.'

Dust explosion risk

In many size reduction operations there are risks of explosion and fire. The liability to dust explosions of many industrial materials has been described by Wilkes.[23]

It is wise to assume that any organic material may form explosive dusts, *i.e.* grain, wood, cork, coal, starch, gums, resin, etc. A number of inorganic materials, especially the metals zirconium, cerium, magnesium and aluminium, are particularly liable to ignite. Iron dusts, and ferro-manganese also have records of fire and explosion. Sulphur has a particularly bad record for fire, but does not seem to explode, nor do graphite and pure carbon seem to explode in air.

Flour and meal milling industries suffer from dust explosions. A mill of 50 cu. ft. capacity would require only 1 oz. of powdered cereal to give the critical minimum concentrations of 0.02 oz./cu.ft. necessary to cause an explosion. All mills, therefore, are liable to contain the necessary concentration of explosive particles. Preventive measures should include avoidance of explosive concentrations and ignition temperatures, and isolation of dangerous processes. Similar information appears in an article by Hartman.[24]

Leclercq[25] has put forward a method of determining the liability to explosion of finely divided material. Excellent extensive research upon this subject is described in two papers by the joint authors, Boyle and Llewellyn.[26] The electrostatic ignitabilities of a number of varied substances in the form of dust clouds or powders were determined under a variety of physical conditions. Under certain circumstances aluminium was an electrostatic hazard as great as magnesium. Ferro-manganese, zinc and silicon dusts were also liable to explosion.

These authors found dangerous concentrations also with tetryl, acaroid resin, polymethyl methacrylate, sugar and flowers of sulphur. Gum, cotton and cordite were also liable to electrostatic ignition.

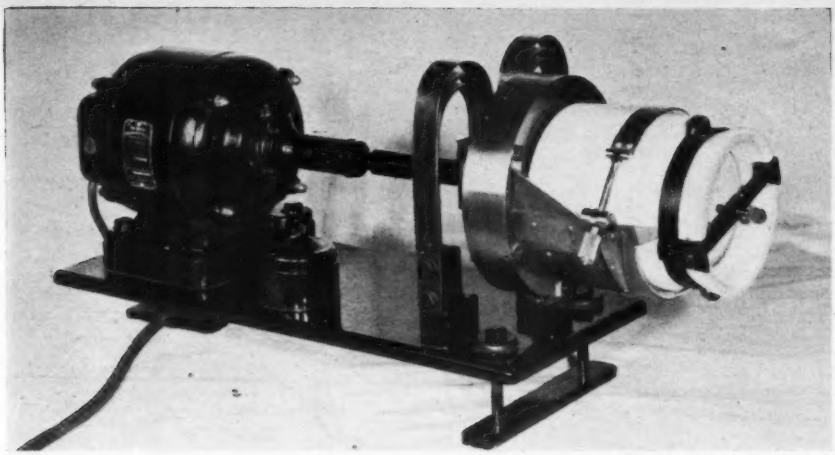
With all combustible material the ease of ignition increased with the reduction in particle size. All particles less than 10μ were vulnerable. The critical concentration for magnesium and aluminium dusts were both in the order of 50 mg./litre. To some extent the critical concentration depends upon the number of particles in the atmosphere. Accurate data are presented as a result of carefully standardised experiments. The papers conclude with good bibliographies.

Methods of rock reduction

The size reduction of hard rock in the mass may be speeded by modern aids to quarrying. War-time demolition methods employing internal shaped hollow explosives giving directional effects have been tested in Swedish iron ore mines.[27] Whilst proving generally more expensive than normal blasting explosives tamped into previously prepared drill holes, the externally placed charges have been shown to be useful for the localising of fragmenta-

TABLE 2. COMPARISON OF HARDNESS VALUES FOR MINERALS

Mineral	*Mohs No.*	*Hardness No. in kg./mm.²*	
		Krushchov	*Dmitriev*
Talc	1	2.4	10— 11
Gypsum	2	36	39— 90
Calcite	3	109	145— 165
Fluorite	4	189	175— 260
Apatite	5	536	550— 690
Orthoclase	6	795	780— 850
Quartz	7	1,120	1,120—1,460
Topaz	8	1,427	1,800—2,000
Corundum	9	2,000	2,050—2,200
Diamond	10	10,060	———



[*Photo: Griffin & Tatlock Ltd.*

Vibratory ball mill for laboratory use.

tion. For certain purposes this reduction of blocks and boulders to more exact-sized primary lumps is an obvious advantage. Another aid is an oxygenated oil flame which, when directed through a blowpipe at the rock face, produces a rapid expansion and resultant spalling of relatively large pieces of rock.[28] The jet piercing process is also described by Hagen,[29] who states that, as applied to ironstone, holes may be made at the rate of 6.3 yd./hr. The face of the stone drilled in this way is shattered by the flame into conveniently sized lumps for transport from the site.

An hydraulic method of breaking off lumps of coal in dangerous mines has been described by Seaman.[30] First a hole is drilled in the coal face large enough to take a round, stainless steel bar which is chambered at intervals to accommodate telescopic pistons. Water under pressure forces out the pistons, thereby causing the cleavage of the spoil into large fragments.

Metal powder production

Numerous references to the production of metal powders of all kinds appear in a private monthly bulletin.[31] One novel method of manufacturing copper powder is due to Hayami,[32] whereby the metal is produced by the interaction of a solution of copper sulphate with waste sulphite liquor. Hansley[33] describes the manufacture of powdered sodium hydride (1 to 10μ) by a reaction between molten sodium and hydrogen containing 0.5% acetylene at 300 to 385°C. in a nitrogen-filled vessel.

New crushing and grinding plant

At the Achema IX exhibition[34] in Frankfurt last year there was a trend towards **electric motorisation of drives** for mills and mixing machines, which were capable of working at supersonic speeds, thereby permitting emulsions to be made more quickly. The finer dispersion greatly improves the efficacy of pharmaceutical and cosmetic emulsions and the taste and tolerance of luxury foods is enhanced.

An especially **small ball mill** of 10 to 75 cc. capacity for mixing and milling 1 to 10 g. of metallic powders is described by Summers-Smith.[35] A **midget swing hammer mill** known as the *Atomill* is powered by a ½-h.p. electric motor running at 10,000 r.p.m.[36] Material is fed to the grinding chamber by a feeder screw, is reduced to the desired size to pass a screen and is discharged into a receiving bin situated below the mill. Provision is made for the inclusion of an inert atmosphere or for cooling air when necessary. All parts are heavily chromium-plated to resist corrosion.

Mikropulverisers[2] are available for disintegrating moist, tacky or unctuous materials such as electrical porcelain clay-body soaps, DDT, moulding compounds containing fibrous fillers, etc. From a grist 1½ in. average diameter, fines –80 mesh are discharged.

Gustafson[37] describes an **air-swept rod mill** whereby the ground mineral perlite may be removed rapidly from the pulverising zone to avoid the formation of fines. A new type of **cement clinker compound mill**, 40 ft. long by 7 ft. 6 in. diameter, is stated to have produced more than 200,000 tons in a little under two years.[38] Interesting claims are made for a modified **pan mill** covered by British Patent 623,820.[39]

For the underground crushing of stowing materials in thin-seam coal mines a quick acting, high-capacity compact machine is required which gives a uniform product and offers protection against tramp iron damage. **The gyratory cone crusher** is said to offer the best compromise.[40] The larger pulverised fuel installations, working on the indirect system, are becoming equipped either with pendulum mills or with tube mills. The smaller direct-feeding units still favour **ball mills and impact crushers.**[41] Cameron[42] describes a **ball mill fitted with screens** for the collection of the coarser fractions in the crushing of carbon for graphite electrodes, but states that this method is being replaced by **air separation methods**. **A coaxial screen** in a ball mill for the recycling of waste fractions is the subject of U.S. patents.[43, 44]

A useful review of sand grinding and milling plant, including pan mills, bowl mills, mixers and turbine mills, has been given by Goffart.[44] Another review of the same type, but on the subject of flour milling is due to Fraser.[45]

In the milling of asbestos rock it is necessary to preserve the staple of the fibre. For this purpose Symons **short-head cone crushers** are used for primary crushing. The final fluffing of the fibre from the rock is by hammer mill.[46]

A rotary bowl type mill has been designed for crushing and mixing plastics.[47]

The crushing of cullet (broken glass), limestone, etc., in glass works is now accomplished by **hammer mills.**[49] It is claimed that these mills consume less power than other mills which have been applied to this purpose. A new type of hammer mill employing **two rotors** is claimed in a British patent by Rudes.[50] **A triple rotary hammer crusher** has been patented by Wright.[51] Another U.S. patent dealing with the hammer mill is due to Lauter.[52]

Improved jaw crushers are described by Ebersol in two U.S. patent applications.[53, 54] The aim of these developments is to provide a two-stage crushing action with independent means for adjusting the discharge gap of each stage. The Blake jaw crusher, having the movable jaw pivoted at the feed end and a removable corrugated crushing plate, has been studied by Gieskieng.[55] Lemmon and Strawn[63] describe a **reversible crusher** with adjustable chute and breaker.

Brant details a **multiple-ball mill** consisting of three parallel shafts on which the ball mills rotate.[56]

Developments in machinery and maintenance methods

Mechanical improvement in machines for all stages of comminution are mentioned frequently in the literature. The abrasive effects of crushed material getting into mill bearings has caused a spate of patents purporting to overcome this trouble. Three claims are made for bearing protection for gyratory crushers.[57, 58, 59] Improvements in the gyratory crusher operating mechanism in which a ball-and-socket joint is incorporated between the eccentric and the straight sleeve mounting are claimed by Kennedy.[60]

The foundations of heavy crushing mills often call for considerable preparation and are consequently costly. One method for overcoming the need for heavy foundations is to suspend the grinding unit, for example a roll crusher, from steel cables supported by light steel columns.[61] The vibration is damped considerably and the wear and tear is reduced. The use of torsion bar springing to obtain the pressure on the crushing plate in a roll crusher is described in a patent specification by R. and J. Moritz.[62]

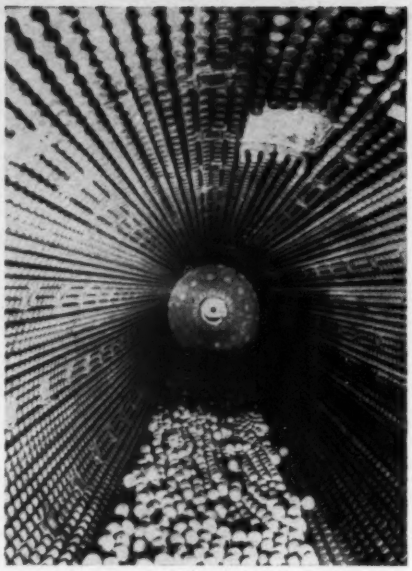
Owing to a shortage of forged manganese steel balls for ball mills in India, white cast-iron balls were employed with success for the grinding of gold-bearing quartz.[64] Similarly scarcity and delivery difficulties have provided the opportunity for more repairs to castings by welding and the use of hard chromium, electro-welded deposits for abrasion-resistant surfaces of gears and pinions for mills of various kinds.[65]

A new type of manganese steel liner plate for ball mills is the *Henricot* stud liner plate.[66] The studs serve as anvils for concentrating the impact of the balls and the depressions between the studs give more grip upon the balls, enabling them to be carried further up the side of the mill before cascading down with increased impetus upon the charge. The reduced working surface area of the liner plate reduces the risk of distortion due to the spreading of the manganese steel under impact.

Tramp iron in swing hammer disintegrators may be eliminated by deflectors which protect the mill from this form of accidental damage.[67]

An oiling system for edge runner mill mullers is described by Pristupa[68] which helped to keep production at a high level. Other practical details of crushing machine operation are dealt with in a paper by Majer.[69]

Finally, there is a fascinating historical survey of crushing and grinding by Lebetter.[70]



[*Photo: Edgar Allen & Co., Ltd.*

Inside of a ball mill lined with 'Henricot' stud liners.

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Chemicals for mothproofing

The progeny from a single female moth might consume 92½ lb. of wool in a year. As long ago as 1921 the world's total loss due to moths was placed at 22,500,000 lb. p.a. In terms of money, the United Kingdom's losses caused by moths has been estimated at £1,000,000 p.a. Today the figure is likely to be very much larger. Indeed, appreciation of the moth menace has grown to such an extent that the British Government has set up a working party to consider mothproofing, particularly with respect to its economic and social implications.

All these facts underline the real need for this book.* Being both a chemist and a textile technologist, Mr. Moncrieff is well qualified to write it.

The first three chapters deal with clothes and house moths, carpet beetles and the nature of the damage caused by wool pests. Then, in eight chapters, Mr. Moncrieff considers the chemicals available for mothproofing—dyestuffs, fluorides, colourless dyestuffs of the triphenylmethane series, Mitin FF, pentachlorphenol, DDT, phosphonium compounds and formaldehyde. In each case the chemistry of the mothproof is discussed, followed by a description of methods of application. The remaining six chapters are devoted to the following subjects: nutritional requirements of the clothes moth larvae, mothproofing by modification of the molecular structure of wool, mothproofing during dry cleaning, infestation precautions and remedies, breeding and rearing moths, and testing the mothproof.

The book is written in an agreeable and easily comprehensible style, and can be recommended to all concerned with moth prevention.

**Mothproofing.* By R. W. Moncrieff. Leonard Hill Ltd., London, 1950, pp. 192, including index, 29 illustrations, 17s.

Construction and Operation of the Gleep Atomic Pile

The Governments of Britain, Canada and the U.S. recently decided to release more information on the design, construction and operation of low-power atomic piles, being satisfied that the information will not materially assist other nations in the development of atomic weapons, since low-power research reactors are unsuitable for this purpose. However, the release of this information does not alter the Atomic Energy Act of 1946 and it is still illegal for private institutions or individuals to construct a reactor unless they are permitted under the Act to obtain the necessary uranium or other fissile material. One of the first results of this new policy in the U.K. has been the release of the following details of Britain's first pile, the Graphite Low Energy Experimental Pile (Gleep), which began operating at Harwell on August 15, 1947. This information, of considerable importance to chemical engineers, is given below as received from the Ministry of Supply.

THE Gleep is a slow neutron reactor using graphite as a moderator and natural uranium and uranium dioxide as fissile material.

It was constructed to meet two main requirements:

(*a*) To run at as high a power as possible without elaborate cooling arrangements. This high power was needed so that radio-active isotopes could be produced in Gleep until such time as the danger Harwell pile (BEPO) became divergent.

(*b*) To compare slow neutron absorption cross-sections of the elements by the pile modulation method.

As a later development it was found that Gleep could be used to provide an accurately known and reproducible thermal neutron flux in the range 10^6 to 10^8 n/cm²/sec. This flux can now be used to determine activation cross-sections.

General description

The pile is built in the form of a right octagonal prism of graphite lying on one of its sides. The reacting core is cylindrical (length 5.24 m., radius 2.86 m.), with the uranium rods lying horizontally in the form of a line lattice of pitch 7¼ in.; the reflector forms the remainder of the octagon, the lower corners of which are filled in with graphite introduced for constructional reasons only. Fig. 1 shows the general arrangement of Gleep.

The total quantity of graphite in the pile is 505 long tons. The graphite is stacked in 40 layers, each layer being built with graphite blocks stacked in such a manner as to resemble a parquet floor. The graphite blocks are of two standard types each measuring 7¼ × 7¼ × 29 in. Various non-standard and half blocks are incorporated, but leading dimensions of all blocks are based upon the fundamental 'pitch' unit of 7¼ in. The edges of some blocks are chamfered, and grooves are cut in other blocks, so that a lattice of diamond-shaped holes of 1.85 in. side runs through the pile from *A* face to *C* face.

The reacting core is loaded with uranium metal up to a radius of 1.75 m., the outer region being loaded with uranium dioxide.

The uranium metal is in the form of cylindrical bars 12 in. long by 0.9 in. diam., and is sprayed with aluminium of 0.0003 in. thickness to prevent the escape of recoil fission products.

In order to increase its density, the uranium dioxide is pressed into pellets 1.60 in. diam. and 2 in. long. These pellets are wrapped in paper containers and inserted in batches of six into aluminium cans of 0.01 in. thickness. This makes a uranium dioxide cartridge 12 in. long by 1.62 in. diam., weighing 2.6 kg.

The Gleep contains 12 tons of uranium metal and 21 tons of uranium dioxide.

There is a 3-ft. air space between the sides of the graphite structure and the inside of the biological shield. This allows access to the inside of the pile if the necessity arises for the removal of any of the uranium cartridges. The concrete biological shield is of 5 ft. thickness around the sides and is 4 ft. thick on the top of the pile.

On the *B* face of the pile there is a hole in the shield which is filled by graphite blocks to form a square section thermal column of 5-ft. side and 7 ft. long. There is a large access hole on the top of the pile so that an additional thermal column can be stacked on the top of the pile if it is required.

There is an elementary ventilation system for the pile capable of delivering 5,000 cu.ft./min. of air. This is provided to remove active argon from the pile, and to provide some cooling of the uranium cartridges. The air is forced by the baffle on *A* face to flow over the uranium cartridges and is extracted by a suction fan on the top of the pile. When the air system is on, it is arranged that the pressure inside the pile is always less than atmospheric; this ensures that there is no leak of radio-active air into the building. The air is ejected through a short stack on the roof of the building (the top of the stack is 60 ft. above ground level) and when the air has diffused to ground level outside the building, its activity is below the tolerance level.

By using the air-cooling system, the pile can be run at a power of 100 kW.

The Gleep has an excess effective reproduction constant of 2×10^{-3} when all absorbers are removed from the pile. This excess k is controlled by four cadmium rods which move together, as shown in Fig. 1. These four rods are known as the coarse control; the single rod shown in Fig. 1 is used as a fine control. All the control rods can be moved up and down by electric motors, which are situated on the outside of the pile and operated from the pile control room.

In addition there are two sets (each consisting of three rods) of emergency shut-down rods. These cadmium rods are held right out of the pile when it is operating, by magnetic clutches. If the pile power rises above a pre-set level, a trip circuit cuts off the current to the magnetic clutches, and the emergency rods fall into the pile under gravity. On the end of a shut-down rod shaft is a disc keyed to the shaft and positioned to rotate between the poles of four electro-magnets to form an electric brake. As the rods approach the 'fully in' position, the magnets of the brake are energised, and the rod motion is retarded to a gentle halt.

To indicate the position of the control rods in the pile, two transmitter magslips, driven through gearing so that one rotates 50 times as fast as the other, are mounted near the drive motors. These transmitters are electrically connected to receivers in the control room, so that direct readings of the positions of the rods are given on two dials. By this means the position of the rods can be read with an accuracy of ± 1 mm.

The power level of the pile is measured

by six ionisation chambers of 5 l. volume, containing boron trifluoride gas at a pressure of 20.7 cm. Hg. Three of these chambers are used for pile control, and the other three are used to operate the emergency shut-down mechanism. All the chambers have pre-amplifiers attached to them, the main amplifiers being in the pile control room. Initially, as the pile power is raised from zero, the resistor in series with a chamber is changed, so that the chamber measures all powers up to 1 kW without any change of position. (The resistor can be changed by operating a wafer switch outside the pile.)

Above 1 kW the chamber is wound out of the pile successively to two pre-set positions, in which the neutron fluxes are factors of 10 and 100 times lower than the flux at the original position.

In this way, with only three positions for each chamber, six decades of pile power can be measured. It will be seen from Fig. 1 that there are eight ionisation chamber holes in the pile. The two additional holes are used for experimental work; one of them is used permanently for work with the Gleep oscillator.

Rough temperature measurements are made at four points in the pile by means of resistance thermometers. Two of these thermometers are strapped on to uranium cartridges near the centre of the pile, one is embedded in a uranium dioxide cartridge, and the remaining one is embedded in a graphite cylinder which has been lowered down one of the vertical experimental holes. The temperatures are recorded continuously in the control room, and it is arranged that if the temperature of the uranium metal cartridges exceeds 60°C., the pile is automatically shut down.

The Gleep has only seven experimental holes, details of which are as follows:

(*a*) Hole J_1, running from face *A* to face *C* through the centre of the pile, 3 in. diam. Since this hole is along the axis of the pile and therefore parallel to the uranium holes, it is used for pile oscillator work (*vide infra*).

(*b*) Hole J, running from face *B* to face *D* through the centre of the pile and thermal column, 3 in. diam.

(*c*) Hole *K*, to one side of the thermal column, running from face *B* to face *D*, 5 × 4 in. The maximum slow neutron flux in this hole at 100 kW is 3×10^{10} n/cm.^{2}/sec., and this hole was used for the manufacture of radio-isotopes when Gleep first began operating.

(*d*) Hole L_1, to one side of the thermal column, running from face *B* to face *D*, 8 × 8 in. This hole can be used for testing the effect on the reproduction constant of the pile of inserting large quantities of material.

(*e*) Hole *W*, running from top to bottom of the pile through the centre, 3 in. diam.

(*f*) Holes *X* and *Y*, running from top to bottom either side of hole *W*, $4\frac{1}{2}$ in. diam.

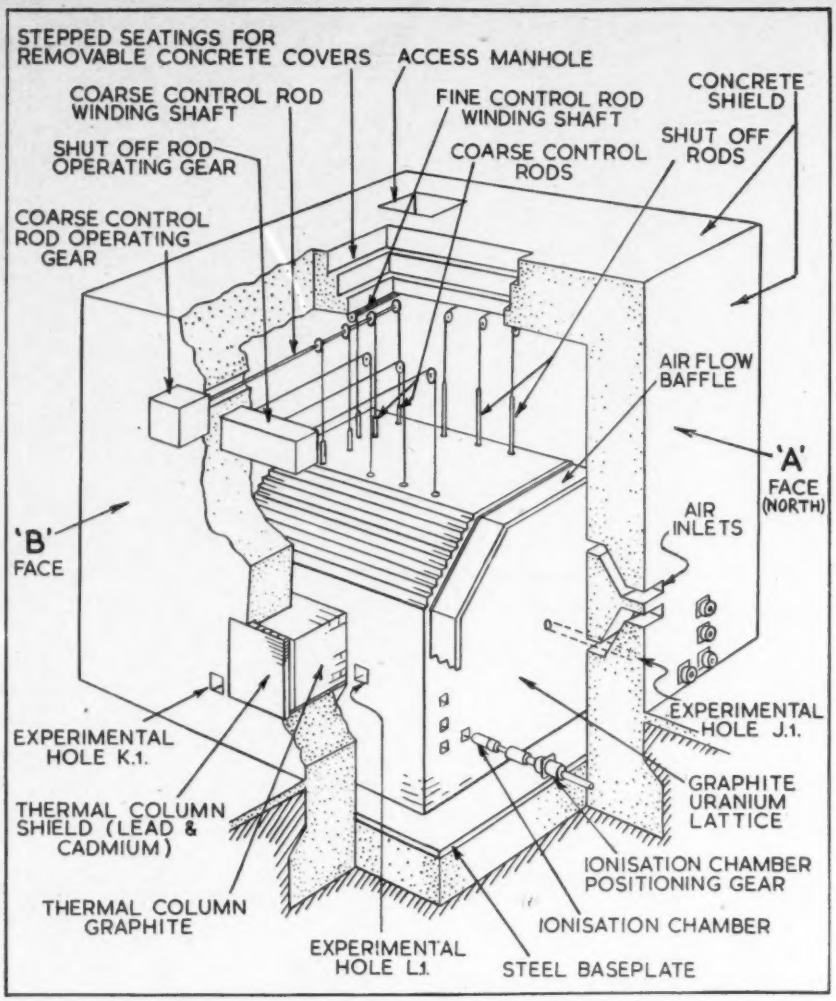

Fig. 1. General arrangement of Gleep.

Although in the foregoing list, holes J_1, J and *W* are all stated to pass through the centre of the pile, they are in fact offset from each other by half of a lattice pitch.

Temperature and pressure coefficients

Soon after Gleep started operating, the pile control rods were calibrated so that the amount of k they took up when in a given position in the pile was known. This was done by first of all balancing the pile with the control rods so that it was running steadily at low power, then withdrawing the control rods a measured amount, at the same time observing the rate of rise of pile power. From a knowledge of the number of delayed neutron emitters, the half lives of these emitters and the rate of rise of pile power, it is possible to calculate the change which has been made in the effective reproduction constant.

Also, shortly after the pile started operating, a run was made at 100 kW without using the air-cooling system. As the temperature of the pile rose, the control rods had to be withdrawn in order to keep the power at 100 kW. From the calibration of the control rods and the measured temperature rise, the temperature coefficient of the pile was deduced.

The temperature coefficient depends on the temperature distribution in the pile, but for a pile running without any cooling, the temperature is, of course, highest at the centre and falls off towards the edges in much the same way as the thermal neutron flux does.

For such a temperature distribution, and for the same change in the graphite temperature as in the uranium temperature at the centre of the pile, the temperature coefficient was found to be:

Change in effective reproduction constant is -2.9×10^{-5}/°C.

It should be noted that the pile has a negative temperature coefficient, and that this prevents the pile from rising to a very high temperature if, for example, the

control system were to fail. In fact, since the excess k of Gleep is only 2×10^{-3}, a temperature coefficient of 2.9×10^{-5}/°C. means that the Gleep could only rise 70°C. in temperature before all the excess k disappeared.

The pressure coefficient of the pile was measured in the following way. The pile was run steadily at low power for about ½ hr., and then the control rods were set in position. Next, with all the holes in the pile shield closed as well as possible, the extractor fan was switched on. This put the pile under 3.5 cm. of water suction (the mean value as recorded by a manometer on either side of the pile), and the rise in pile power was observed for 15 min.

From the rate of rise of pile power the pressure coefficient was calculated, and the answer obtained was:

Change in effective reproduction constant is: -6.5×10^{-6}/mb.

This coefficient is also negative, since an increase in atmospheric pressure increases the amount of nitrogen in the pile and reduces its reactivity.

High power running of the pile

In order to check that no dangerous amount of activity was escaping from the stack at high power running, a 20-atm. argon chamber was installed in the top of the stack to measure the gamma activity of the effluent from the pile. During the initial runs at high power, it was noticed that not only did this chamber give a higher current than would be expected from the argon 41 activity, but also that this activity built up with time when working at a constant power level. This suggested that fission product activity was escaping from the uranium in the pile, and the following experiments confirmed this.

An aluminium tube was placed through the $\mathcal{J}$ hole of the pile. Arrangements were made to flow air through this tube and then over a Geiger-Müller counter. With the pile running at constant power, a given flow of air was passed through the system and the background counting rate due to the argon 41 was determined. A small foil of bare uranium of known area was then put into the centre of the tube and the counting rate due to the fission products carried off from the foil obtained. This bare foil was then replaced, firstly by samples of the aluminium-sprayed uranium rod, and secondly by a uranium dioxide cartridge. The results of this comparison of bare uranium with the two types of cartridge used in the pile showed that, whereas the aluminium spraying was 99.5% efficient, the uranium dioxide cartridges were leaking gaseous fission products into the air stream at a very high rate.

In view of these results, the channels of the pile containing the uranium dioxide cartridges were blocked off from the air stream. Since they form the outside ring of the pile where the neutron flux (and therefore the heat output) is low compared with the central channels, this did not in fact make the oxide cartridges rise appreciably in temperature when running at 100 kW.

The discharge of fission products with the associated build-up of activity on the stack monitor was reduced by a factor of 10.

Experimental work

Most of the experiments described in this section were done with the Gleep oscillator, and so an account of this oscillator is given first.

The oscillator takes small samples rapidly from the edge of the pile to the centre of hole $\mathcal{J}_1$. These samples are moved in and out of the pile with periodic motion, and this induces a periodic modulation in the power level of the pile. This periodic modulation in the power level is measured by a boron trifluoride ionisation chamber, is amplified and the resulting signal integrated. The integrated signal is a measure of the change in k produced by a sample.

The samples are housed in a graphite container and can be driven from the outside of the pile shield to the centre of the pile by means of an electro-mechanical system. The samples are moved between two positions in a regular manner so as to produce a periodic modulation of the pile power (the samples remain in each position for 30 sec. and take 3 sec. to move from one position to the other).

In the electrical apparatus used in the measurements the main control unit governs the motion of a sample in the pile. The current from the boron trifluoride ionisation chamber consists of two parts:

(*a*) A steady current, which produces a voltage across the resistor in series with the chamber. This voltage is balanced by a 'backing-off' voltage, which is a measure of the mean chamber current.

(*b*) A periodically varying current due to the periodic modulation of the pile by a sample. The voltage that this produces across the resistor is amplified by the d.c. amplifier.

The voltage output from the amplifier is multiplied separately by sine and cosine factors of the same periodicity as the motion of the sample (this is done by having sliders moving with simple harmonic motion across resistor cards connected to the output of the amplifier), and is then fed into two velodyne integrators and integrated for one complete period.

The integrated answers are thus proportional to the two fundamental components in the Fourier analysis of the pile modulation. If we consider only the first harmonic components in the pile modulation, it may be shown that the percentage modulation (the actual depth of modulation divided by the mean power level) produced by a sample is proportional to the change in k it would produce if placed at the centre of the pile. Thus the integrated answers are a measure of change in k.

Re-equipment of a barytes mine

At present one-quarter of the total British production of barytes comes from Scotland. The Muirshel mine, near Loch Winnoch, Renfrewshire, now one of the largest producers in the United Kingdom, has been modernised and current production amounts to 1,000 tons per month. A description of the exploration, development and modernisation work is given in the *Mining Journal* (1950, **135** (5999), pp. 134-135).

Barytes seems to have been mined at Muirshel from the middle of the 18th century until 1920, although on a small scale, the entire production up to 1920 amounting only to some 18,000 tons. Since then the property has been closed down and it was not until 1942 that the mining rights were again acquired. In 1945 the Muirshel Barytes Co. Ltd. was formed, under which name the mine is now operated. The new company's operations have shown that the earlier miners did little more than scratch the surface of the massive veins which exploration has since revealed. In the last seven years over 40,000 tons of barytes have been taken from the mine. Reserves actually blocked out and proved are sufficient for many years' work at the current rate of production. The present development and diamond drilling programmes have already increased the estimated life of the mine by many more years, while further interesting possibilities have yet to be fully explored. When completed, the programme of modernisation and re-equipment at present being carried out by the Muirshel Barytes Co. Ltd. will make the mine one of the best equipped in Britain.

Situated at approximately 1,100 ft. above sea-level in bleak moorland country, the mine generates its own electricity supply, the equipment comprising two 70-kW National engines and a 30-kW Ruston engine. Compressed air for the drilling is supplied by a 65-h.p. Blackstone engine and an auxiliary electric set, the drills used being Climax, Ingersoll Rand and Consolidated Pneumatic. The pumping equipment includes two Mono pumps at the 210 ft. shaft and a Maclay and a Ruston pump at the 110 ft. shaft. The capacity of the Mono pump is about 50 gal./min. and those of the Maclay and Ruston pumps 100 and 80 gal./min., respectively. Heavy Bedford shovels are employed throughout the mine.

The barytes extracted is fed through chutes into mine cars of 1-ton capacity and hoisted in a 1-ton skip. Up to 1920, the spar was dressed at a local grinding mill. Nowadays it is loaded on to motor lorries at the mine and conveyed by road either to Glasgow or to the railway station at Loch Winnoch for dispatch to other parts of Britain. No grading, dressing or sorting is done at the mine.

Oxygen Lance Production of Stainless Steel

By W. H. Everard and D. J. O. Brandt, A.R.S.M., B.Sc., A.I.M.

The use of gaseous oxygen to accelerate the refining process in steel-making has probably made the most progress among electric steel-makers, both in the U.S.A. and Great Britain. Towards the end of 1947 this method was adopted experimentally by Edgar Allen & Co. Ltd. of Sheffield. Since then, the production of stainless steel from 100% scrap charges using the oxygen lance has been a matter of regular routine. The process is described in this article,* which includes a discussion of the chemistry involved.

THE use of the oxygen lance for refining a high-chromium steel, as opposed to its use in non-chrome steels, enables the heat to be worked at a temperature some 200°C. higher than normal. Oxygen rapidly attacks the oxidisable elements, and the very high heat of formation of SiO_2 and Cr_2O_3 (or CrO) can raise the bath temperature to well over 1,800°C. (estimated) in a few minutes. Feeding the bath with iron oxide does not give the same results; the reactions are much slower, since the oxide (ore or scale) must be first melted and absorbed into the bath.

The higher the bath temperature the less stable are the oxides of chromium, manganese, iron and silicon, and the more stable is the oxide of carbon, CO. In an oxidised bath the formation of carbon monoxide will therefore be enhanced and the formation of the metallic oxides repressed, so that the carbon may be removed with a reduction in the losses of chromium (and manganese) which are normally unavoidable in ore-refining practice. Owing to the losses of chromium into the slag, making it extremely thick and heavy at normal steel-making temperatures, it is virtually impossible to refine a 100% stainless scrap charge with an ore boil. With oxygen, and the extra 200°C. bath temperature available, this is now an everyday practice.

When ore or scale is fed into a furnace in which there is a slag containing oxides of chromium, some of it may be 'passivated' owing to attack by Cr_2O_3 forming a hard refractory layer of the double oxide, $FeO.Cr_2O_3$, on its surface, thus hindering the attack on the carbon in the bath.[3] With oxygen this does not occur.

The chromium-bearing slag obtained in the oxygen-refining of stainless steel can be reduced by ferro-silicon, and an appreciable portion of chromium oxidised during blowing may be recovered and returned to the metal. Thus, the addition of virgin chromium in the form of low-carbon ferro-chromium is cut down to a minimum.

Practical experience has shown that all these features, together with the fact that during oxygen refining the electrodes in the arc furnace may be raised and the current cut off, mean that the economies gained will amply offset the expenditure necessary for the oxygen. The production of stainless steel by this process is rapidly becoming a standard practice on both sides of the Atlantic.

*Extracted from a paper in the *Journal of the Iron and Steel Institute*, August 1950.

Details of operation

A 4-ton Heroult-type tilting electric arc furnace, with a transformer of 750-kVA capacity is in use at Edgar Allen & Co., for the oxygen refining of stainless steel (Fig. 1). Electrode pillars are located opposite the taphole, and the furnace is worked through two side doors opposite each other. At present, this furnace is equipped with a monolithic dolomite hearth and side walls, and a silica roof.

The oxygen lance consists of naked 20-ft. lengths of standard $\frac{3}{4}$-in. wrought-iron tube and is introduced through the taphole. The lance end is held just below the metal surface and is moved slowly forward during the blow as the pipe is consumed. Oxygen is fed to the lance through $\frac{3}{4}$-in. diam. rubber hose. A liquid oxygen evaporator and storage-tank arrangement have been installed.

Technique of blowing

During the initial experimental stages it was found that the removal of carbon and the life of the lance were improved the higher the rate of flow of oxygen into the bath. Gas is now fed to the bath at a rate of 438 cu.ft./min., and at an initial pressure of 150 lb./sq.in., which is not allowed to fall below 120 lb./sq.in. The operation requires control to prevent a violent reaction which may damage the furnace refractories.

At the commencement of blowing, the lance is inserted through the taphole and an observer is stationed at one of the side doors of the furnace. The oxygen is then turned on and the lance end pushed just below the metal surface at about 30° to the horizontal (see Fig. 2). If the bath shows a tendency to rise dangerously, blowing ceases for a few minutes to allow the bath to subside. Blowing finally ceases when the inlet pressure falls below 120 lb./sq.in. or, in the case of the second blow, when the bath goes dead. The behaviour of the bath appears quite arbitrary; sometimes it is possible to make a continuous blow using the first 2,000 cu. ft. and at other times it may be necessary to adopt the above intermittent practice.

Two types of slag are obtained by varying the method of introduction of the lance. If kept in the steel there is a more rapid erosion of the lance and a bulky and dense slag is formed; on the other hand, if the lance end is in the slag the latter becomes more fluid and 'watery.' The former method is more satisfactory because the thick slag 'blankets' the heat from the top refractories, and the addition of scrap is not needed to cool the slag. The reason for this variation in the slags is not yet properly understood.

Pipe consumption averages 17 ft. for the first blow and 15 ft. for the second blow, *i.e.* 0.90 ft./100 cu.ft. and 0.83 ft./100 cu.ft. respectively. This is probably due to the improvement in the cooling action with the faster gas flows.

Oxygen blowing is characterised by the formation of fume and by the appearance of flames at the various exits in the furnace structure, *i.e.*, doors, electrode holes, etc. During the first few minutes fume is predominant; as the reaction gets under way, it is replaced by a large, hot, bright, yellow flame.

Summarising, the practical aspects of the process present no particular difficulties apart from the, at present, unavoidable wear on the dolomite hearth at the high temperatures involved; oxygen should be fed into the bath at a rate as fast as is consistent with safety and at a pressure of not lower than 120 lb./sq.in. Lance life and chemical efficiency are improved by a faster rate of oxygen flow.

A chromium recovery of 83% on all scrap charge is obtained; the oxygen consumption averages 1,080 cu.ft./ton tapped, and power consumption 630 kWh./ton. Overall recovery is 93%.

Chemistry of refining stainless steel with oxygen

One of the principal difficulties which at present stand in the way of a clearer appreciation of the chemical mechanism of this process is the fact that the temperatures involved are necessarily very high. Owing to the high heats of combustion, the oxidation of chromium and, in particular, of silicon, forces the bath temperature into ranges which cannot be

measured with the normal type of Pt/Pt-Rh immersion pyrometer. An understanding of the chemical mechanism of the refining process is thereby hindered.

The work of Hilty[1] in the U.S.A. has demonstrated that, in a bath of chromium-bearing steel under the action of the oxygen lance in the laboratory, there is a well-marked relationship between the chromium/carbon ratio in the bath and the bath temperatures. The results recorded in Hilty's laboratory experiments show a logarithmic relationship between Cr/C and temperature. If it is assumed that conditions in the electric arc furnace under the action of the oxygen lance are reasonably similar to conditions in Hilty's laboratory-scale experiments, then an estimate of the bath temperature during oxygen injection on Edgar Allen's 4-ton furnace may be made from the Cr/C ratio of the steel, extrapolating where necessary.

This device has been resorted to in all cases where the bath temperature is higher than an immersion thermocouple can record. It must be stressed, however, that these temperatures are only estimates, and liable to error.

It is generally assumed that the stable oxide of chromium is the chromic oxide, Cr_2O_3, and that this compound exists in the bath under oxidising conditions. Hilty, however, has suggested the existence of the lower oxide CrO, in the steel, which may be wholly or partly converted to Cr_2O_3 in the slag. Further evidence on this point has been submitted by Chen and Chipman,[2] Clark,[3] and Körber and Oelsen.[4] The latter state that in acid slags the lower oxide CrO definitely preponderates.

At the start of oxygen injection at Edgar Allen & Co. Ltd. the metal is free of slag; this builds up during the oxidation due to erosion of the bottom and banks of the furnace. It may thus vary in both bulk and composition within wide limits and, under present conditions, it is impossible to state whether at any time it is more acid than basic or *vice versa*. Slag samples are not drawn during refining and, as they are difficult to dissolve, their analysis is not straightforward.

The figures for reaction efficiency were calculated on the assumption that all the chromium is oxidised to Cr_2O_3 in the slag. This does not deny the possible existence of CrO in the metal, but in any case the actual quantity of chromium existent in this form would be extremely small. Oxygen equivalents and silicon equivalents are calculated on the basis of the equations:

$$4Cr + 3O_2 = 2Cr_2O_3,$$

$$\text{and } 2Cr_2O_3 + 3Si = 4Cr + 3SiO_2$$

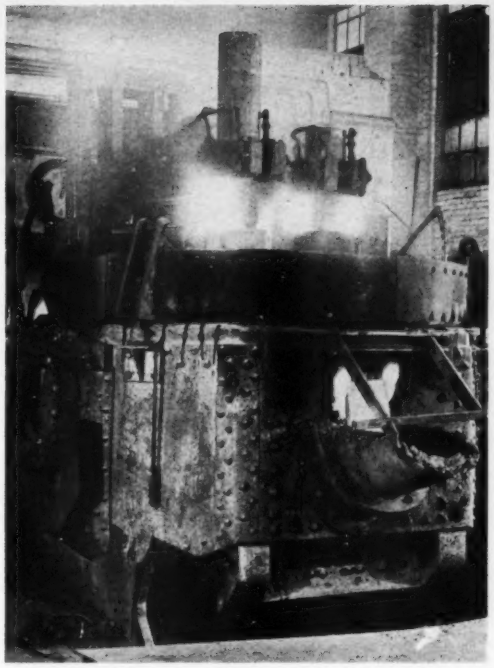

Fig. 1. Four-ton Heroult furnace used for the production of 65-cwt. stainless heats with oxygen.

Results of first blow

The following facts emerge from an examination of results:

Efficiency.—Between 50 and 70% of the oxygen blown oxidises alloying elements. High initial silicon and carbon contents tend to give the best efficiency figures.

Effect of silicon.—A high initial silicon content (due to silicon in the scrap charged), exerts a protective action on the carbon, chromium and manganese. The affinity of silicon for oxygen at these temperatures is roughly the same as that of carbon, and markedly greater than that of manganese and, therefore, provided that these constituents are present in sufficient concentrations to be equally active, the silicon will tend to be oxidised first.

Temperature increment. — The temperature rise during blowing, as estimated by the Hilty formula, appears to be very large dependent on the silicon and chromium oxidised, which is to be expected, as these two oxides have the highest heats of formation.

Oxidation of manganese.—Manganese is also protected by silicon and, consequently, although the initial manganese content may be noticeably high, the removal of manganese may be relatively insignificant. The rapidly increasing temperature and the considerable quantity of carbon left in the steel also inhibit the oxidation of manganese. As for chromium, increasing temperature stabilises manganese in contradistinction to carbon; thus a high temperature enables a high Mn/C ratio to exist in the bath.

Oxidation of iron.—The degree to which iron has been oxidised can only be estimated by the specific consumption. It appears that, in general, between a quarter and a half of the total volume of oxygen blown is consumed in the oxidation of iron.

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Sulphur, phosphorus and nickel.—At steel-making temperatures, oxides of these elements are less stable than the oxides of iron, silicon, carbon, chromium and manganese, and are therefore not removed by oxygen. The increases in sulphur and phosphorus during blowing are partly due to the effect of concentration since the weight of the bath decreases, and partly to the absorption of these elements by the furnace lining from previous heats.

Results of second blow

In the second blow there are two important changes—increased temperature and reduced silicon content. The most noticeable effects of these are the reduced temperature increments and the increased degree of oxidation of the chromium. There also appears to be a slight general improvement in the efficiency of blowing, which may be due to the higher temperatures attained.

With the removal of silicon the equilibrium is between the oxides of carbon, manganese and chromium only. The absence of the silicon causes the chromium to be more easily oxidised, in spite of the higher temperature tending to enhance the oxidation of carbon and inhibit the formation of chromium oxides. In the case of manganese, however, the increased temperature has a more marked effect, and the oxidation of manganese in the second blow is actually less than in the first. The general tendency towards the greater retention of manganese in the second blow, compared with the faster oxidation of the chromium in the same period, is evident in all the heats.

Apart from these points the behaviour of the steels in the second blow is generally similar to that in the first.

Reduction with ferro-silicon

The reduction of chromium from the basic slag after blowing is at present extremely difficult to control, since the volume and composition of the slag are variable.

About 30 to 50% of the available silicon is used in the reduction of chromium, the reduction of manganese accounting for about another 3%; 20% is unchanged and enters the metal, and maintains equilibrium with the slag which absorbs SiO_2 from the oxidation of the ferro-silicon. About 50% of the silicon added is oxidised by the furnace atmosphere or by reduction of iron oxide in the slag.

The chemistry of these reactions is as follows:

Reduction of chromium:

$$2Cr_2O_3 + 3Si \rightarrow 4Cr + 3SiO_2$$

Reduction of manganese:

$$2MnO + Si \rightarrow 2Mn + SiO_2$$

Reduction of iron:

$$2FeO + Si \rightarrow 2Fe + SiO_2$$

(*and reduction of higher oxides*)

'Fixing' of silica:

$$SiO_2 + \text{free } CaO \rightarrow xCaO.ySiO_2$$

The degree of completion of these reactions in the slag/ferro-silicon reduction process will depend on the activities of the various reactants or, expressed another way, on the concentrations of free oxides present (chromium, manganese and iron oxides) and the concentration of free CaO in the slag necessary to fix the silica and to avoid an undesirably high silicon content in the steel. The effect of temperature should only be marked in so far as it affects the activities of these oxides, since silicon has a higher affinity for oxygen than has Cr, Mn, or Fe at all temperatures.

It is clear that high temperatures do, in fact, appear to assist good chromium recoveries. The recorded temperatures are only estimated after the second blow, the real temperatures being probably much lower, due to radiation and conduction losses and to the addition of 3 cwt. of scrap when required.

A second factor which appears to influence the efficiency of the reduction process is the quantity of silica already in the slag. Where a heat starts with a high silicon metal, the SiO_2 in the slag depends on the extent of hearth erosion. If the slag after the second blow is already rich in silica it will probably be unable to absorb much more, and the series of reactions causing reduction of the chromium oxide will be checked. Silicon then either enters the bath unchanged or is oxidised atmospherically without corresponding slag reduction. A high initial silicon content may therefore protect the chromium in the oxidation period at the cost of preventing its subsequent recovery during reduction.

The slag

The following slag compositions were obtained in one heat:

	SiO_2 %	Fe %	Al_2O_3 %	CaO %
Melting slag* (reducing conditions)	14.82	2.24	17.04	45.09
After first blow ..	18.93	5.15	7.32	27.72
After second blow	11.74	4.87	6.33	22.38
After ferro-silicon reduction ..	21.55	7.23	4.30	27.82

Fig. 2. Commencement of oxygen blowing with observer stationed at side door of furnace.

	MgO %	MnO %	Cr_2O_3 %	TiO_2 %
Melting slag* (reducing conditions)	8.39	0.85	1.01	8.91
After first blow ..	6.80	3.21	27.19	1.04
After second blow	4.85	3.27	42.71	0.23
After ferro-silicon reduction ..	10.79	2.47	22.16	0.17

*Discarded before oxygen injection.

Working on the chromium balance, the weights of the last three slags are, by calculation, 807, 1,635 and 2,050 lb., respectively.

The increase in the slag bulk during the reduction period, as well as during the oxidation period, is shown by the CaO and MgO figures to have come from the hearth. The silica balance (excluding possible roof drip) confirms this. The apparent increase in the iron content of the slag during reduction is directly contrary to physico-chemical theory which predicts that, since oxides of iron are less stable than either the oxides of chromium or manganese, the reduction of iron should take place prior to that of Mn and Cr. The fact that this is apparently not so may be explained by the possible 'fixing' of the iron oxides either by CaO in the form of the calcic ferrites or by SiO_2 in the form of iron silicates.

This explanation is tentative only, and more slag data are being collected.

Conclusions

The chemical mechanism of the oxidation of carbon, silicon, chromium and manganese from a bath of stainless steel with an oxygen blast is at present only partially understood. The results available from Edgar Allen & Co. Ltd. show that the way in which oxidation will proceed during blowing depends upon two main factors—starting temperature and initial silicon content. Silicon exerts a 'protective' action on both carbon and chromium and enables the temperature to be raised rapidly. This is an advantage initially but may be offset later by the difficulty of reducing the chromium from a slag high in silica, at any rate with ferro-silicon. The use of aluminium might prove an advantage in these cases. With the accumulation and examination of further data, a clearer understanding of the principles involved should become possible; work on these lines is proceeding.

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Silicone grease

Development of lubricating greases particularly designed for special equipment employing shielded or sealed bearings and operating under wide temperature ranges has been described in a new patent (U.S. 2,508,741). These greases contain silicones which offer a marked advantage over mineral oils in that they possess a comparatively uniform viscosity over a wide range of temperatures and are not susceptible to evaporation loss as a comparable mineral oil. Synthetic silicone greases heretofore available have one undesirable characteristic—lack of resistance to shear. Under high-shearing stresses such as ball or roller lubrication the structure breaks down and their consistency falls off until they are liquid or semi-liquid. The present compositions, it is claimed, overcome this characteristic by the use of special acid components of the metal soaps.

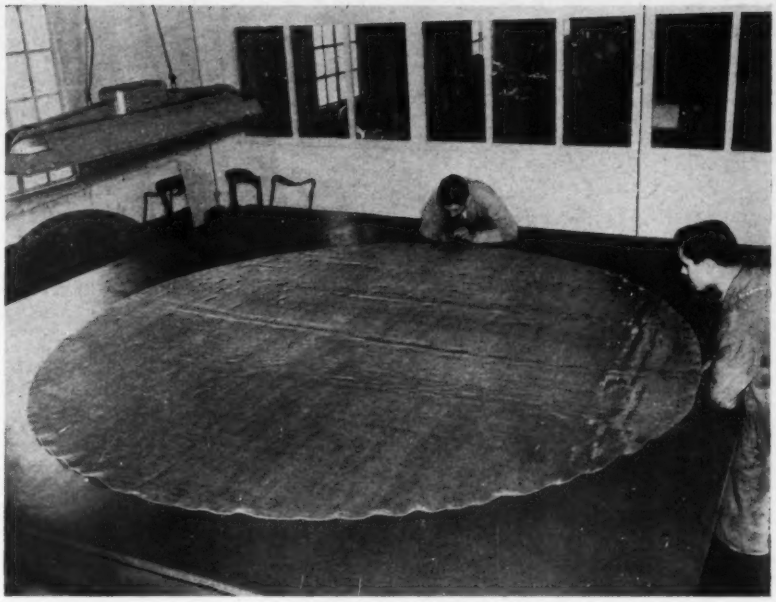
The metallic constituent of the soap may be any of the metals such as sodium, lithium, calcium, barium, aluminium, etc. A typical product obtained is a smooth-light-coloured buttery grease having the following calculated composition:

	%
Lithium soap	12.8
Glycerin	0.9
Mineral oil	13.4
Dimethyl silicone polymer ..	72.9

Such a grease possesses a fairly high dropping point, is low in evaporation loss and low in oil bleeding and also meets torque requirements at –67°F. In addition, its grease breakdown test performance is said to be outstanding, since it shows the product to behave excellently up to 300°F. without breakdown or change in texture of the product.

Noble Metal Catalysts

FOR several years the large-scale industrial application of noble metal catalysts was confined to contact process sulphuric acid plants, which by the year 1920 had required several hundred thousand ounces of platinum, chiefly in the form of platinised asbestos. Although the importance of platinum in the sulphuric acid industry has receded somewhat since the development of efficient base metal catalysts, other industrial processes have since come into use requiring platinum, rhodium, palladium and silver in considerable quantities, and the number and volume of chemical products manufactured by their aid are continually increasing.



Rhodium-platinum gauze for a 3-m. diam. ammonia burner under inspection in the Johnson Matthey catalyst shop.

Platinum catalysts for ammonia oxidation

The oxidation of ammonia by its passage mixed with air over heated platinum sponge was first observed by Kuhlmann in 1839, but it was not until 1902 that Ostwald's British Patent Specifications 698 and 8300 disclosed the conditions necessary to obtain in practical operation a useful yield of product. The first technical plant, which was erected at Gerthe in Westphalia during 1909, was based on Ostwald's design, and had a number of small chambers arranged to provide heat exchange between incoming and outgoing gases fitted with catalyst pads formed of pure platinum foil. Although the Ostwald catalyst is still in use in two European factories, the method of using superposed gauze nets introduced by Kaiser in Brit. Pat. 24035/1911 has become the most general system.

The abundant supply of low-cost pure ammonia resulting from the development of the processes of ammonia synthesis led to a tremendous expansion of the ammonia oxidation industry, particularly in the period 1920-30, and it is now the source of almost the whole world's output of nitric acid. Although base metal oxide catalysts have been brought into use during periods of platinum scarcity, no catalyst as efficient as the platinum gauze pad has been developed, and the process remains fundamentally that which Ostwald devised.

Oxidation at elevated pressure

The most important modification to the process has been the introduction by the du Pont Co. of oxidation at elevated pressure, usually 6 to 7 atm.[1, 2] By this method the conversion of nitrogen oxides to nitric acid is greatly facilitated, but the efficiency of oxidation is reduced so that to obtain a satisfactory yield it is necessary to work at higher temperatures than in an atmospheric pressure plant.

The increase in operating temperature is accompanied by a reduction in the life of a platinum gauze catalyst, which is very marked under the conditions necessary for high-pressure operation. Research which was stimulated by this circumstance led to the introduction of rhodium-platinum alloys containing up to 10% of rhodium, which have a considerably lower high-temperature loss rate than has pure platinum.[3] At the present time gauze woven from 10% rhodium-platinum wire is practically universal in high-pressure plants. It is arranged in multi-layer catalyst pads of 20 to 30 individual nets, which enable a very large quantity of ammonia to be oxidised in a relatively small converter.[4]

Atmospheric pressure operation

The benefits gained by the presence of rhodium in the catalyst diminish as the working temperature is lowered, and in plants working at atmospheric pressure a lower content of rhodium, generally about 5%, is now customary. While pressure oxidation is extensively used in the U.S.A., Great Britain and Empire countries, it has not been much adopted on the Continent. Oxidation at atmospheric pressure, sometimes followed by compression of the oxidised gas before absorption, is generally preferred.

In some cases air enriched with oxygen or an oxygen-steam mixture may be employed as the oxidising medium.[5] The search for operational economy and thermal efficiency has led to the use in the atmospheric pressure plants of large diameter burners: 1, 1.5 and 2 m. diam. circular burners are common.[6] Those most recently built have a diameter of nearly 3 m. In weaving the very large areas of fine mesh gauze of uniform quality needed to clothe these large converters, a high degree of skill and technical experience is necessary.

TABLE 1. STANDARD GAUZE CATALYSTS FOR AMMONIA OXIDATION
(British dimensions)

Composition	*Mesh per lin. in.*	*Wire diam. (in.)*	*Approx. weight (oz/sq.ft.)*	*Available form*	*Use*
Pure platinum	80	0.0026	1.40	Rectangular or circular 4-ply welded pads*	In nitration process sulphuric acid plants
Pure platinum	80	0.0030	1.84	Rectangular or circular seamless nets up to 42 in. diam. with reinforced edges	In nitric acid plants employing atmospheric pressure oxidation
5% rhodium-platinum	80	0.0030	1.78	Ditto	Ditto
10% rhodium-platinum	80	0.0030	1.72	Ditto	Ditto (when operating at high-loading ratio)
10% rhodium-platinum	80	0.0030	1.72	Circular or hexagonal nets with reinforced edges	In pressure oxidation plants

*Can be additionally reinforced by stitching with platinum wire

Gauze catalysts for nitration process sulphuric acid plants

Most nitration process sulphuric acid plants are supplied with nitrogen oxides by means of a small oxidation unit.[8] These units are generally of the ' United Alkali ' pattern with a built-in heat exchanger, fitted with a pad of four rectangular gauzes mounted in the vertical plane. The process requires flexibility in the supply of the oxides and, whereas the ammonia burners of a nitric acid plant generally operate under almost constant load, these small units are worked at capacities ranging from less than 50% to more than 200% of normal. Gauze temperatures are relatively low and, since pure platinum is an effective catalyst over a wider range of conditions than are the rhodium-platinum alloys, it is preferred for this type of work.

The catalysts are supplied as made-up pads of four reinforced gauzes welded together at the edges, and are available in sizes to suit all makes of plant. A special gauze woven of wire 0.0026 in. diam. is used for this purpose, since the lighter material is found more satisfactory in these small units. If required, the gauze may be additionally reinforced by stitching with platinum wire or by welding across the surface, but these procedures are not generally considered necessary.

Improvements to the catalyst gauze

Because of the economic importance of the ammonia oxidation process, it has been the subject of intensive investigation, and there are numerous patents relating to improvements in the composition and method of fabrication of gauzes. Of the many alternative materials, the ternary alloys of rhodium, palladium and platinum are probably the most promising,[7] but in general the established range of rhodium-platinum alloys, in plain weave, has not yet been surpassed for all-round efficiency, low loss rate and length of effective life. It is necessary, however, that the most careful control over the purity and metallurgical conditions of the alloy is maintained at every stage of manufacture.

Platinum catalysts for the contact process . . .

In the equilibrium system containing sulphur dioxide, sulphur trioxide and oxygen, the proportion of sulphur trioxide diminishes with increasing temperature, so that the oxidation reaction must be conducted at the lowest practicable temperature. This requires a very active catalyst, which must be dispersed on the surface of a carrier, or must be sufficiently bulky in itself to allow time for the considerable volume of gas passing through the catalyst bed to reach equilibrium.

In practical working only two types of catalyst, finely divided platinum on the surface of a carrier and variously prepared active vanadium compounds, are currently in use. Because of their availability through plant manufacturers in robust forms of guaranteed efficiency, the proprietary vanadium catalysts have been widely adopted, particularly in sulphur-burning plants of the hot-gas type. For metallurgical gas plants which must necessarily have provision for through gas cleaning, platinum catalysts have advantages in their ability to maintain conversion efficiency when fed with an inlet gas of varying composition, and the use of platinum is retained in a substantial number of ore gas plants.[9]

The effectiveness of a platinum contact mass is greatly influenced by the nature of the carrier, and silica gel is now generally used for this purpose, the dispersal of the

TABLE 2. STANDARD GAUZE CATALYSTS FOR AMMONIA OXIDATION
(Metric dimensions)

Composition	*Mesh per sq.cm.*	*Wire diam. (mm.)*	*Approx. weight (kg/sq.m.)*	*Available form*	*Use*
Pure platinum	1024	0.06	0.415	Rectangular or circular 4-ply welded pads*	In nitration process sulphuric acid plants
Pure platinum	1024	0.06	0.415	Circular nets, 1, 1.75, 2.1, 2.9 m. nominal diam. with reinforced edges†	In nitric acid plants (Bamag and Uhde types) as backing gauzes
3% rhodium-platinum	1024	0.06	0.410	Ditto	In nitric acid plants (Bamag and Uhde types) as intermediate gauze, or where low rhodium content pad is preferred
5% rhodium-platinum	1024	0.06	0.400	Ditto	In nitric acid plants (Bamag and Uhde types): this form is most generally used
3 or 5% rhodium-platinum	3600	0.04	0.370 or 0.366	Circular nets 1, 1.75 and 2 m. nominal diam. with reinforced edges	In nitric acid plants (Bamag and Uhde types): not generally recommended because of high cost and greater fragility

*Can be additionally reinforced by stitching with platinum wire.
†Sizes up to 2.2 m. nominal diam. without seams are available.

TABLE 3. PLATINUM CATALYSTS

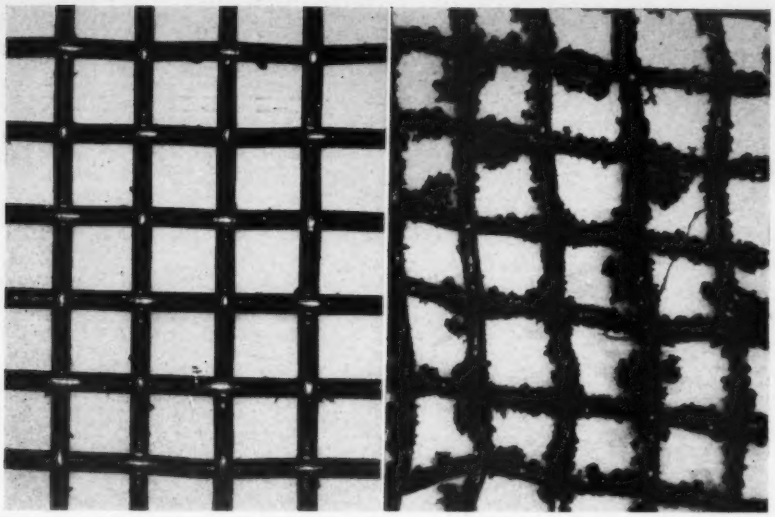
Catalyst	*Available form*	*Use*
Platinum and rhodium-platinum gauze	See Tables 1 and 2	Oxidation of ammonia
Platinum sponge	Pure ignited sponge, 99.98%	Analytical determination
Platinum dispersed on carriers	On silica gel, asbestos, quartz and pumice	Gas-phase catalytic reactions
Platinum dispersed on carriers	On charcoal	Liquid-phase catalytic reactions
Unsupported dispersed platinum	Platinum black, platinum oxide (Adams' catalyst)	Liquid-phase catalytic reactions
Platinum salts	Ammonium chlorplatinite, chlorplatinic acid	Preparation of supported catalysts

TABLE 4. PALLADIUM CATALYSTS

Catalyst	*Available form*	*Use*
Palladium gauze	Plain woven, wire diam. 0.004 in. and upwards	Gas-phase catalytic hydrogenation
Palladium sponge	Pure ignited sponge	Gas-phase catalytic hydrogenation
Palladium dispersed on carriers	On silica gel, asbestos, quartz and pumice	Gas-phase catalytic hydrogenation
Palladium dispersed on carriers	On charcoal	Liquid-phase catalytic hydrogenation
Unsupported dispersed palladium	Palladium black, palladium oxide	Liquid-phase catalytic hydrogenation
Palladium salts	Ammonium chlorpalladite, palladium chloride	Preparation of supported catalysts

TABLE 5. SILVER CATALYSTS

Catalyst	*Available form*	*Use*
Chemically pure silver gauze	99.99% pure 20 mesh/lin. in., 0.014 in. wire diam.	In the vapour-phase dehydrogenation of alcohols
Chemically pure silver gauze	99.99% pure 4 mesh/linear in., 0.04 in. wire diam.	For supporting silver catalyst beds
Crystalline silver, activated	99.99% pure crystal, selected particle sizes from 5 mm. downwards	In the vapour-phase dehydrogenation of alcohols
Silver nitrate	Pure crystallised	Preparation of supported catalysts
Silver oxide	Pure dry powder	Preparation of dispersed catalysts



Rhodium-platinum gauze, before use and as returned for recovery. Metal of the highest purity is necessary to avoid early embrittlement and fracture through the structural change which occurs during the catalytic process.

active catalyst being usually an integral part of the process of making the gel.[10] The platinum is introduced into the mass as an aqueous solution of ammonium chlorplatinite or platinic chloride.

. . . and for liquid-phase reaction

Although the manufacture of nitric and sulphuric acids requires the largest quantities of platinum, it has also a considerable use as a hydrogenation catalyst in organic synthesis. For this purpose it is dispersed on the surface of carriers such as pumice or charcoal, or it may be used directly in the form of platinum black without a support. The form most extensively used for liquid-phase hydrogenation is the easily reducible oxide known as Adams' catalyst, which is now manufactured for this purpose on a fairly large scale.[11]

Palladium catalysts

Palladium has a greater affinity for hydrogen than has any other metal, and is a very active hydrogenation catalyst. For example, a mixture of hydrogen and acetylene at atmospheric pressure and at a temperature of about 200° C., passed over a catalyst of palladised silica gel, produces ethylene in good yield with negligible cracking to ethane. This process has been developed on a large scale, and plants producing 5,000 tons of ethylene per month were erected in Germany.[12]

Palladium is at present mainly used as a liquid-phase hydrogenation catalyst dispersed on pumice or charcoal, or used unsupported as palladium black. The available forms and methods of preparation are similar to those of the corresponding platinum catalysts.

Some use is also made of palladium as a catalyst for the purification of oxygen and hydrogen, and for the removal of residual oxygen from atmospheres required for bright annealing and other operations where complete freedom from oxygen is necessary. Palladium gauze is occasionally used for this purpose, but palladised pumice or palladised silica gel are more usually employed.

Silver catalysts for the dehydrogenation of alcohols

The catalytic vapour-phase dehydrogenation of both methyl and ethyl alcohols are processes of considerable importance. The great volume of formaldehyde solution consumed industrially is almost all manufactured by the dehydrogenation of methyl alcohol, while the parallel process with ethyl alcohol is a major source of acetaldehyde.

Copper has been used to some extent as a catalyst for both reactions, but it is less efficient for this purpose than silver, which is now almost universally employed. The process in either case requires the passage of a stream of water vapour, air and an excess of alcohol vapour through a catalyst bed maintained by the heat of reaction at about 600° C. The products after condensation are fractionated and the small proportion of unconverted alcohol is recycled.[13, 14]

A little alcohol is lost through side reactions, and very careful control of temperature and composition is necessary to keep this at a minimum rate. The efficiencies of the conversion have been improved considerably since the processes were first developed, partly through the adoption of new types of catalyst, and through an increase in catalyst purity. In one or two instances, the silver catalyst is dispersed on a carrier. For this purpose pure recrystallised silver nitrate is supplied. In most existing installations, however, the catalyst is either a thick pad of many layers of silver gauze, or a shallow bed of crystalline silver. In either case metal of very high purity is necessary and the wire for gauze manufacture requires both close chemical and metallurgical control in its preparation.

Seamless circles with plain edges are normally supplied. Circles over 24 in. diam. are seamed. The crystalline catalyst is prepared by electrodeposition under conditions regulated to produce hard crystals which are screened into close size ranges.

Other catalytic processes employing silver

Silver is an active catalyst for many oxidation reactions, and its use in a considerable number of vapour-phase oxidations has been examined. For example, its use in the preparation of methacrylonitrile and acrylonitrile has been described.[15] Attention has been principally directed, however, towards the aerial oxidation of ethylene, and plant has been constructed in Great Britain, the U.S. and Germany. A plant was projected in Germany having an output of 100 tons/month of ethylene oxide. The silver catalyst was dispersed on pumice and was prepared by impregnating the pumice with silver oxide paste, and then reducing this oxide in a nitrogen/hydrogen mixture. The total silver content of the catalyst mass would have been about 120,000 oz.[16]

Ruthenium, osmium and iridium catalysts

Although these metals in various forms have from time to time been mentioned in technical publications and patents as being suitable catalysts for a number of processes, there has so far been no large-scale development in their use and they are not considered standard industrial catalyst materials.

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Petroleum Production in England

Although England is not normally connected with oilfields, no less than 600,000 tons of crude petroleum were obtained from English workings between 1938 and 1949. Besides yielding oil, the work has brought to light hitherto unknown potash and coal deposits. Here is a brief account of English oil production.

NATURAL petroleum has been worked on a small scale in England for centuries, but the possibility of the occurrence of oilfields was neglected until the 1914-18 war. At that time supplies of petroleum from overseas were threatened and a serious search was sponsored by the Government. A number of wells were drilled, but the results were disappointing. Prospecting ceased at the end of this war, but geological interest in the project remained.

In 1936 an extensive drilling programme was launched and from 1938 to 1949 600,000 tons of crude oil and petrol were obtained from English workings.* Although this represents an average annual production of 50,000 tons, an output of 112,760 tons was actually achieved in 1943. An account of these explorations is given in 'Petroleum in England,' a booklet recently published by Anglo-Iranian Oil Co. Ltd., whose subsidiary, D'Arcy Exploration Co. Ltd., have been responsible for the major drilling programme and, in fact, achieved the first big success by discovering the Eakring oil field.

Investigations were carried out in Sussex, Dorset, the Midlands, west Lancashire, Nottinghamshire, Yorkshire and other counties. The D'Arcy Exploration Co. tested the possibilities of Permian gas and coal by drilling a 5,040-ft. well at Eskdale. This yielded 2,500,000 cu.ft./day of nearly pure methane. Up to the present this has not been used, but remains available for industrial use. Substantial potash deposits were also discovered. In the summer of 1939, a shallow oilfield was found at Formby in west Lancashire within a few days of the discovery of larger and important fields at Eakring in Nottinghamshire. At Formby, 47 shallow holes have been drilled for production and information, of which eight still produce by pumping under vacuum and, after ten years, yield about 17 tons/month with a cumulative total of 8,035 tons at the end of 1949. The oil is similar in gravity to the Eakring crudes and has a very low petrol-oil ratio.

The crude oils from the millstone grit sands of the Eakring district are dark brown, but the limestone crudes have a greenish tinge. The motor and aviation petroleum distillates have predominant naphthenic characteristics, and show high octane values. The paraffin wax content is high, being about double that of average mid-Continent crudes. The sulphur is low, being 0.01% in the petroleum fraction, and there is practically no elemental sulphur in the crude. The limestone oils contain about 0.4% by weight of sulphur, as against approximately 0.1% in the grit oils. High-grade lubricating oils are obtainable from all these crudes.

*General statistics of the British petroleum industry appear on page 41.

Equipment

In the early stages, elaborate and costly well equipment and layout involved excessive labour and considerable loss of time. The outbreak of the war caused shortages of both materials and labour and this situation coincided with the planning of intensive drilling operations. Modern American methods, introduced at this time, led to great improvements in overall speed and cost.

A pumping unit is installed on every completed production well and most equipment is driven electrically. Although the petrol-oil ratio on the fields is small, the well-head gas is collected from the annulus between the casing and production tubing in each well and from the water separators in the Eakring end of the field. Formerly 160,000 cu. ft. of gas were treated each day, but the figure has now fallen to 50,000. The conservation of the valuable casing-head gas was considered early in the life of the field and a charcoal absorption plant was installed. This recovers the pentane and heavier fractions, giving a petroleum which can be used directly in the company's vehicles without such difficulties as vapour locking (1,000 cu. ft. of gas yield just over 1 gal. of petroleum).

Water separators

In the early life of the field the oil from the crestal wells was water-free, but as drilling proceeded down flank and as production increased, some wells began to produce water with the crude. As the number of wells producing water increased, it was found necessary to install locally designed and constructed water separators at each collecting station. These separators were heated by steam coils and packed with wood wool to increase separating capacity. A centralised water-separating plant, however, now replaces these individual plants and comprises a battery of three direct-fired heater-treaters. The direct heating feature, by which the oil-water mixture is heated by passing over the fire tube of the furnace incorporated in the treater, is very economical in the petrol used as fuel. Wet oil from the various fields is now pumped to this centralised plant for treatment, which reduces the water content of the crude to about 1%. The separating process is assisted by the addition of a chemical emulsion breaker to the crude at the well-head.

The high wax content of the oil causes many difficulties, both in surface lines and in the upper part of the wells, as wax starts to settle out at 80°F. In the surface lines this is overcome by pumping a steel plunger with end leathers through all lines at suitable intervals. In the wells the wax accumulation lies in the top 700 to 800 ft. and the need to pull tubing for mechanical dewaxing has been overcome by electrical heating.

Attempts to raise output

There have been many experiments to increase production from the sands, including the use of wetting agents. Well shooting is the only one which has given commercial results, the sustained production capacity of individual wells having on the average been more than doubled. Polar blasting gelatine is used, being exploded by time-bombs. Charges up to 400 lb. are lowered in containers, with boosting pellets distributed throughout the charge. The present practice is to use cement tamping above the explosive. Acid treatment has given useful results in limestone producers.

A fairly rapid fall in reservoir pressure with production was experienced in most sands, due to an absence of water drive in the formation, and if allowed to persist would have resulted in the ultimate production of a comparatively small proportion of the original oil in place. Accordingly towards the end of 1947 injection of water was started and by the middle of 1948 some 60,000 gal./day of water were being injected into five wells. By November 1948, oil production at the Eakring field had increased to 43 tons/day from a production of 28 tons/day prior to the effect of the water drive. Moreover, the higher rate of production has been maintained and marginal wells have produced as much as ten years' production at the pre-injection rate and are still producing several times as much oil as formerly.

A by-product of the search for oil in England has been the large amount of new information, of both economic and scientific importance, which has resulted from the geological and geophysical surveys and exploration drilling. Apart from the location of an important deposit of potash (see INTERNATIONAL CHEMICAL ENGINEERING, 1950, **31** (11), p. 492), an extensive new coalfield has been discovered in Lincolnshire, as well as an extension to the Nottingham coalfield. In the south, geological surveys have indicated areas where more concealed coalfields may await discovery. There is every hope that the oil search will produce as many by-products in the future as it has in the past, and that the ultimate amount of oil will greatly exceed the total achieved to date.

New Plant and Equipment

Metal-ceramic materials

Under severe thermal conditions conventional ceramics generally fail to combine all the desirable properties such as high strength and resistance to creep and thermal shock. Considerable interest has therefore been shown in bodies in which metals and ceramics are intimately associated to constitute what is virtually a new class of materials.

The Morgan Crucible Co. Ltd. are developing metal-ceramic materials for many applications. *Metamic* products show good strength and resistance to mechanical and thermal shock at high temperatures, *i.e.* above 800°C. They are made by powder metallurgy techniques and are therefore limited at present in size, with dimensions in inches rather than in feet. Cold strengths (modulus of rupture) will probably be of the order of 10 tons/sq.in., and it is hoped that strengths at high temperatures will reach this figure.

Numerous combinations of metal, ceramic and the methods of manufacture will permit a wide variation in the properties and fields of uses of the new material; thus, for particular uses, one may desire resistance to oxidation, corrosion, erosion or creep, the formulation being varied to meet these requirements.

New fire extinguisher

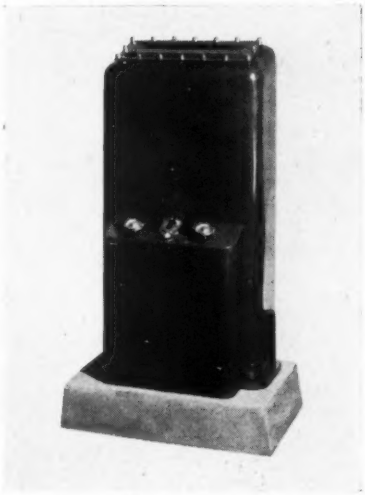
The remarkable speed and effectiveness of a new dry chemical fire extinguisher produced by the Pyrene Co. Ltd. was demonstrated to the technical Press recently. The charge of the extinguisher consists of a CO_2 gas cylinder and a metal canister containing 20 or 30 lb. of the powder. The powder, it is understood, is basically sodium bicarbonate in a very finely divided state, plus other ingredients, the nature of which is not disclosed.

The extinguisher was tested to prove its efficacy against the newer spirits and solvents which are being increasingly used in the chemical and other industries. In the first test, six trays measuring 4 ft. by 2 ft. and containing respectively petrol, butyl alcohol, acetone, di-isobutylene, toluol and isopropyl alcohol were set alight. When all six were blazing fiercely, the extinguisher was brought into action and the whole conflagration was put out completely in a matter of seconds. Three other similar fires were started and these also were extinguished with the remaining charge of the same appliance, it being possible to cut off the powder at will. In a second test, a metal tray, 7 ft. by 4 ft., containing methanol was ignited and put out with another extinguisher of the same type. As a contrast to the solvent fires, a tank of 100 sq. ft. area containing fuel

Tackling a 100 sq. ft. oil fire with the new dry chemical extinguisher.

oil was fired and allowed to burn for some 30 sec. This, too, was put out with the dry chemical extinguisher within seconds. Finally, a running petrol fire, possibly the most serious to deal with, was completely quenched in a matter of seconds.

The new appliance is very much faster than the usual foam-type extinguisher. Whereas, however, the latter appliance covers the burning area with a layer of foam which prevents subsequent ignition, the new powder extinguishes only the immediate blaze, leaving a residue which can be fired. However, as a fast and effective fire killer the appliance has many applications in industry.



Air-cooled ozoniser for water treatment marketed by Paterson Engineering Co. Ltd. Ozone is generated by passing air between an electrode and a dielectric. The treated air is subsequently entrained with the water to be processed.

Self-regulating alternators for small power plants

Despite the extensive development of electrical power networks, small engine-driven A.C. generators are still required where no other supply is available, or to supplement, or as an alternative to a mains supply. Engine-driven generating plant has proved indispensable as a means of reducing peak load demand from the general power system and maintaining supply during power cuts.

To obtain the maximum efficiency from the generators and the electric motors, lighting equipment, etc., supplied, the voltage should be controlled within the limits that ensure the most favourable operating conditions. Voltage can be regulated automatically by supplementary equipment, but to save cost and complications many small generating plants are not equipped with automatic regulators, so that there is likely to be an appreciable reduction in the overall efficiency of the plant under different loading conditions.

To obtain the advantages of automatic voltage regulation without the additional cost of special regulating equipment, a self-regulating alternator has been developed. This alternator, marketed under the name of *Magnicon*, is now being manufactured under licence from the Macfarlane Engineering Co. Ltd., Glasgow, by Crompton Parkinson Ltd., London, W.C.2.

The standard *Magnicon* controls its own voltage within $\pm 2\frac{1}{2}\%$ of the normal value, but machines can be designed with an inherent regulation within $\pm 1\%$. It is available for a wide range of ratings.

Acid-resistant goggles

Non-inflammable, acid-resistant, one-piece goggles have been marketed by Safety Products Ltd. The plastic frame is claimed to be strong and supple and moulds comfortably on to the contours of the face. Available in three sizes with clear, light-green, dark-green and bronze screens, the goggle has a simple press-stud system which enables the screen to be changed quickly and easily when pitted. The frame is perforated, allowing good ventilation but preventing the entrance of dust.

Portable pump

A 57-lb. portable pump, claimed to have an output of 15,000 gal./hr., has been developed by the McCulloch Motors Corp., Los Angeles 45. The pump is a centrifugal type with a 28-ft. suction lift, and is powered by a 5-h.p. petrol engine. It features a non-clogging impeller mounted directly on the engine shaft.

Pump and engine are mounted on a special spring base to ensure good footing on various types of terrain. A centre balance handle adds to the unit's portability. The pump has a kick-proof automatic-rewind starter, a rainproof ignition system and an automatic governor which controls its speed at all loads. A three-way valve allows the use of a separate fuel tank when the pump is being used for long periods of unattended operation.

Labelling and marking device

Drum-coat is a new non-mechanical method of printing letters, numerals, trade-mark devices, etc., on to all types of cylindrical containers by means of a film of rub-proof and weather-resistant ink which is rolled up on a slab and transferred by hand rolling on to a type forme, consisting of a simple bed to hold pre-cast composition letters or numerals. This can be secured at floor level, over which large, heavy drums can be rolled without effort, or at bench height for smaller containers. A special type-casting unit can be supplied for preparing the pre-cast relief-printing surface in any descriptive matter required, or, alternatively, single letters and numerals can be supplied to fit into the forme. This forme can be easily adjusted to accommodate any desired combination of marks, including lettering, numbers, trade-mark devices, etc. By using two or more formes, inked separately, different colours can be marked on the containers simultaneously. For marking flat surfaces, hand-operated formes can be supplied to which the composition type is fixed and, after inking, these formes can be used to give a sharp, clear impression on to the ends of drums or on to flat-sided containers. Makers are Parsons, Fletcher & Co. Ltd.

Decarbonising solution

A new decarbonising solution for all internal combustion engines has been developed by Jenolite Ltd. The solution is supplied ready for use and can be used in any type of steel container. The temperature at which it operates is between 75 and 90°C. The approximate period of immersion is 20 min.

Should any carbon deposits remain after immersion in the decarbonising solution, these are easily removed by wiping with a dry cloth. The components should also be given either a methylated spirit or white spirit rinse, to ensure that no traces of the solution remain after treatment. It is claimed that none of the materials normally used in internal combustion engines, such as iron, steel, bronze, etc., are attacked by the decarbonising process.

The process is said to be non-corrosive and the fumes given off during the operation are harmless. If operators' hands are constantly in contact with the solution, it is recommended that rubber gloves should be worn. The solution has the effect of drying the skin. As an alternative to wearing rubber gloves, a good barrier cream should be used.

Free samples for testing purposes can be obtained from Jenolite Ltd.

Drum marking equipment.

Corrosion testing meter

A multi-combination meter for electrolysis, corrosion and cathodic-protection testing has been introduced in the U.S. by M. C. Miller, New Jersey. It is small, compact and weighs only 13 lb., although it has a low range of 2 mV and special mirror scales 3.9 in. long. The accuracy is 1%.

It is claimed that with this meter practically all measurements encountered in electrolysis and corrosion investigations and cathodic-protection testing in field and laboratory can be made. By use of a circuit selector switch, the two high-sensitivity d.c. instruments can be connected into a variety of measuring circuits for measurement of potentials, current, resistance and soil resistivity. Internal batteries with switch and coarse and fine controls can be used to supply and control current for test purposes. Voltmeters, or voltmeter and ammeter can be used separately or simultaneously. Polarity reversing switches are provided for both current and potential measurements.

Transformer welder

A new small compact arc-welder, designed to cover a wide range of applications, uses an automatic hot start control with a hermetically sealed gas-filled, time-delay magnetic switch that has no open contact. The manufacturers, Air Reduction Co. Inc., New York, state that it has no delicate relays, rectifiers, fuses or open arcing contacts. A special safety feature is the silicone insulation.

New Plant and Equipment

Fill in the Enquiry Coupon on page 44 for further details of the equipment and plant described in

INTERNATIONAL CHEMICAL ENGINEERING.

Single-unit polarograph

Polarography is a rapid and accurate technique of chemical analysis in which the electrolysis of solutions between a dropping mercury cathode and a mercury pool anode is studied. During the electrolysis, a voltage which increases at a uniform rate is applied to the cell and the current/voltage curves thus obtained are recorded.

From a study of these polarograms, the character and concentration of the substances in solution can be determined. The concentration is found from the current increase, *i.e.* the step height. The nature of the substance is indicated by the voltage at which the corresponding step occurs.

A single-unit polarograph in a polished mahogany case has been introduced by Tinsley (Industrial Instruments) Ltd. This instrument has a vibration-proof d.c. amplifier, exchangeable batteries and pen recorder unit. The multiple ranges cover 0.1 to 100 μA.

The unit is operated from 200/250 V, a.c., 50 c/s mains and is supplied complete with batteries, charts, ink, pens and electrode glassware.

Processing kettle for resins

A new type of processing kettle, particularly suited for oil bodying and alkyd resin manufacture, has been developed by Blaw-Knox Co., U.S.A. The apparatus can be fired by either oil or gas and does not require the use of an indirect heat-transfer medium. The furnace temperature approximates that of the kettle, rather than the temperature of the heat source, so that the flow of heat automatically stops when the burners are cut off. This permits rapid cooling and exact temperature regulation.

Photoelectric furnace control

A photoelectric viewing head, a relay control unit and an electrically operated fuel-supply valve form the three basic units of a new system for cutting off the fuel supply to an oil burner instantly upon flame extinction, obviating the risk of explosion. The flame is observed through a 1½-in. diameter aperture in the furnace front and failure of the flame causes the photo-cell immediately to operate the control unit which closes the fuel valve. The relay unit may be as much as 30 ft. away from the viewer.

When flame failure occurs in oil-burning installations, a highly explosive mixture of air and gases rapidly accumulates in the combustion chamber before the fuel supply can be cut off by a manually operated valve. Numerous disastrous explosions have occurred, causing loss of life and destruction to property, due to an attendant attempting to re-ignite the burner before dangerous gases have dispersed. This fully automatic photoelectric fuel-control system for oil-fired installations is supplied by Elcontrol Ltd.

Meetings

Institution of Chemical Engineers

January 13. Midland Branch. 'Application of Vapour-Liquid Equilibria to Extractive Distillation,' by F. H. Garner and S. R. M. Ellis, 3 p.m., Latin Theatre, the University, Edmund Street, Birmingham.

January 19. Graduates' and Students' Section. 'Plant Management in the Dyestuffs Industry,' by P. M. Shaw, 6.30 p.m., Caxton Hall, London, S.W.1.

February 6. 'Liquid-Liquid Extraction,' Introduction by A. S. White. Part 1, 'Flooding Rates in Packed Columns,' by F. R. Dell and H. R. C. Pratt. Part 2, 'Hold - up/Pressure Drop in Packed Columns,' by R. Gayler and H. R. C. Pratt. Part 3, 'A Study of Droplet Behaviour in Packed Columns,' by J. B. Lewis and H. R. C. Pratt, 5.30 p.m., Burlington House, London, W.1.

Society of Chemical Industry

January 16. Chemical Engineering Group. 'The Application of High-Speed Centrifuges in the Chemical and Process Industries,' by R. H. Dolton, 5.30 p.m., Burlington House, London, W.1.

February 5. London Section. 'Recovery of Bromine from Sea Water,' by Dr. R. O. Gibson, 6.30 p.m., London School of Hygiene and Tropical Medicine, Keppel Street, W.C.1.

Institute of Petroleum

January 10. The I.P. Safety Codes. Papers by members of the I.P. Engineering Committee: 'General Considerations,' by E. J. Sturgess; 'Their Application to Production,' by H. de Wilde; 'Their Application to Refining,' by Dr. F. Mayo; 'Their Application to Marketing,' by R. L. Sarjeant, 5.30 p.m., Manson House, 26 Portland Place, London, W.1.

Institute of Fuel

January 15. 'Drying of Coal and Lignite,' by Dr. W. R. Chapman and L. W. Needham; 'Drying of Peat,' by H. N. S. Miller; 'Drying of Sand,' by Dr. E. Seddon; 'Drying of Ores' (author not named); 'Drying of Foundry Moulds and Cores,' by G. T. Hampton; 'Drying of Road Stone and Aggregates,' by L. C. Hopkins, 6.30 p.m., King's College, Newcastle.

January 30. 'Drying Technique,' by R. R. Clegg; 'Instrumentation of Driers,' by W. H. Chipperfield; 'Heating by Electrical Elements,' by J. T. Sharples; 'Drying of Air and Gases,' by Birlec Ltd., 5.30 p.m., Institution of Mechanical Engineers, London.

Institute of Metals

January 16. 'The Production of Some of the Rarer Metals,' by A. R. Powell, 6.30 p.m., University College, Metallurgical Department, Singleton Park, Swansea.

January 25. 'Oxygen in Steelmaking,' by D. J. C. Brandt, 6.30 p.m., James Watt Memorial Institute, Great Charles Street, Birmingham.

January 25. 'Cast Corrosion-Resisting Alloys,' by M. M. Hallet, 6.30 p.m., Grand Hotel, Sheffield.

British Association of Chemists

January 31. Display of scientific films, 7.15 p.m., Wellcome Research Institute, 183 Euston Road, N.W.1 (in conjunction with Scientific Film Association).

Royal Society of Arts

January 17. '1851-1951; A Century of British Industry,' by Lord McGowan, (chairman, I.C.I.), 2.30 p.m., John Adam Street, Adelphi, London, W.C.1.

Manchester Metallurgical Society

January 31. 'Metallurgy of Titanium,' by J. Preston, 6.30 p.m., Engineers' Club, Albert Square, Manchester.

Leeds Metallurgical Society

February 1. 'Recent Developments in Metallurgy,' by E. C. W. Perryman, 7 p.m., Chemistry Department, University, Leeds.

Liverpool Metallurgical Society

February 1. 'Electrodeposition for Engineering Purposes,' by A. W. Hothersall, 7 p.m., Lecture Theatre, Electricity Service Centre, Whitechapel, Liverpool.

Institute of Welding

January 10. 'Application and Developments with Resistance Welding,' by R. Bushell, 7 p.m., Reynolds Hall, College of Technology, Manchester.

Society of Leather Trades Chemists

January 27. 'The Chemist's Part in Accident Prevention,' by Dr. Thierry; 'Development of Control in Vegetable Tanning,' by G. Cooper, 2 p.m., Engineers' Club, Albert Square, Manchester.

Royal Institute of Chemistry

January 11. 'Design and Operation of Laboratory Fractionating Columns,' by L. J. Williamson, 7.30 p.m., Royal Station Hotel, Hull.

January 31. 'Men and Machines,' by Prof. R. E. Lane, 6.45 p.m., lecture theatre, gas showrooms, Town Hall, Manchester.

Hull Chemical and Engineering Society

January 23. 'Centrifuges,' by D. G. Gillies, 7.30 p.m., Church Institute, Hull.

February 6. 'Recent Advances in Gas and Fuel Industry,' by Prof. A. L. Roberts, 7.30 p.m., Church Institute, Hull.

Chemical Society

January 26. 'A Chemist in Industry,' by R. S. Lumsden, 5 p.m., Chemistry Department, United College, St. Andrew's, Scotland.

Recent publications

Zinc and spelter. The story of zinc and its gradual recognition as a metal is described in 'Zinc and Spelter—Notes on the Early History of Zinc,' published by the Zinc Development Association. Among the subjects discussed are the use of zinc ores for the manufacture of brass in the ancient world, the isolation of the metal in China and its introduction in Europe. Although intended primarily for research workers in the history of metals, less specialised readers should find it of interest.

Aluminium alloy castings. The Aluminium Development Association Bulletin No. 17 deals with aluminium alloy castings. It is chiefly addressed to the user with the prime object of helping him to gain the fullest advantage from the properties of aluminium alloys. Methods of casting and notes on the design of castings are included.

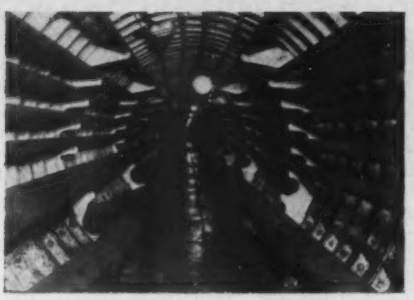
Welding research. Full details of work being carried out by the British Welding Research Association is given in their annual report, 1949-50.

Solvents. A leaflet dealing with *Catarex* solvents 15-9 for oils, fats, waxes and most types of synthetic resins has been published by Petrochemicals Ltd. These solvents are products of the *Catarole* cracking process.

Steam boilers. A fully illustrated booklet has been published by Davey, Paxman & Co. Ltd., to describe their wide range of industrial steam boilers.

Tinplate handbook. The steel-making and tinning processes used in the manufacture of both hot-dipped and electrolytic tinplate are summarised and the factors which determine the suitability of the various types of tinplate for any particular application are discussed in 'Tinplate Handbook,' by W. E. Hoare. The major uses of the principal grades of tinplate are indicated and methods of testing mechanical properties, coating thicknesses and lacquer adhesion are briefly described. The trade terms used by the British and American tinplate industries are defined and methods of grading and classifying the finished tinplates are reviewed. Gauge tables and conversion tables covering a wide range of coating weights and thicknesses are included and an English-French-German-Spanish glossary of technical terms of the trade is given. The book is obtainable free from the Tin Research Institute, Greenford, Middlesex.

Scrubbers. Impingement-type mist eliminators based on the original Calder-Fox design are now available in a wide range of sizes for disposal of practically any suspended mist which might be produced by chemical and physical processes. A leaflet published by Kestner Evaporator & Engineering Co. Ltd. gives technical details of the Calder-Fox gas scrubber which can be constructed in almost any contact material, *e.g.* lead, rubber, *Keebush* or stainless steel.



Pipes being installed in the 9-ft. diameter industrial tunnel under the River Tees.

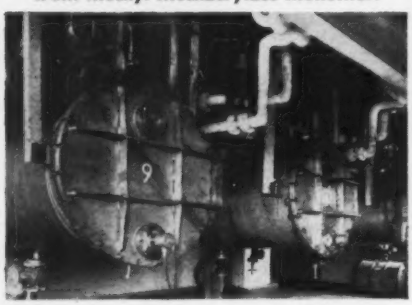
Wilton grows up

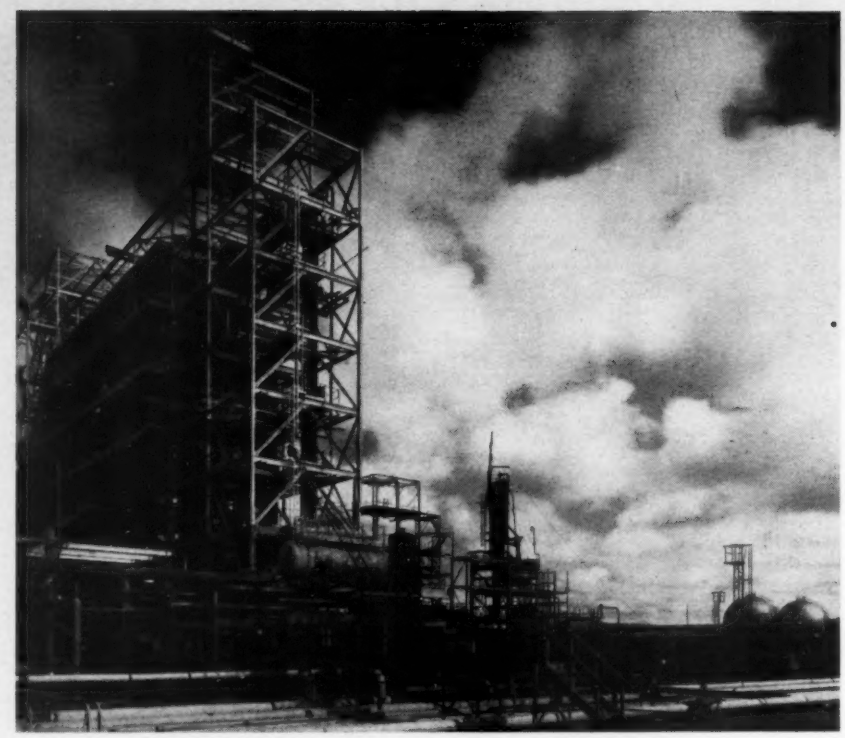
THE biggest project in British chemicals, and certainly among the biggest industrial projects in the world, is Wilton Works, the mammoth chemical complex which Imperial Chemical Industries Ltd. are building on 2,000 acres of the north Yorkshire countryside, hard by the River Tees. Wilton is designed to provide services and raw materials for a whole range of I.C.I. factories which may be similar only in that they make use of the same basic products. It will provide accommodation for new projects, such as the *Terylene* synthetic fibre plant reported in our December issue, and also for over-flow plants from I.C.I.'s 11 divisions.

Wilton was formally inaugurated in October 1949 (see I.C.E., Nov. 1949), when two plants were in operation—one producing phenol formaldehyde moulding powders, the other *Perspex* sheet. Now, the first stage of construction, estimated to cost £22,000,000, is well under way, as shown in the photographs. Among the most important plants in the final stages of building is the petroleum cracker which will convert crude oil into ethylene, methane-hydrogen, propylene and butane. Among the most spectacular of Wilton's features is a 1,750-ft. tunnel under the Tees which will carry the 10 miles of pipelines linking the works with the other great I.C.I. factory at Billingham.

There are now 3,500 workers on the Wilton site; 3,000 on construction and 500 manning the plants already working. When the first stage is finished, it is believed Wilton will give work to 4,500 people.

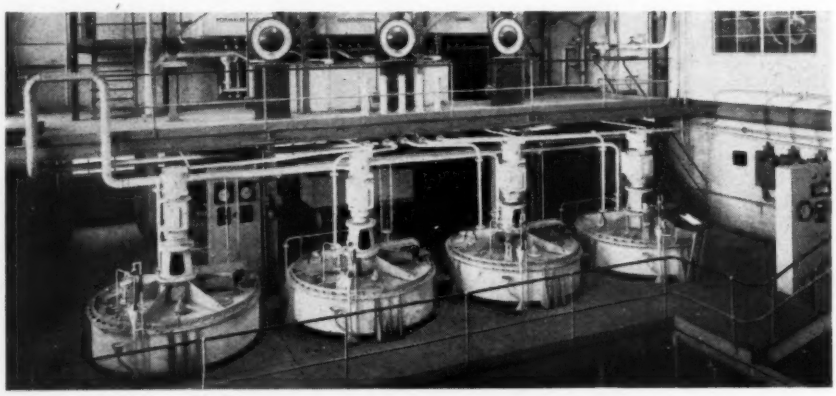
Part of the *Perspex* sheet plant, showing vessels used in an intermediate stage of manufacture from methyl methacrylate monomer.





Part of the oil-cracking plant, looking east.

'Piccadilly Circus,' a busy intersection of transport routes, around which many plants are situated.



Part of the phenol formaldehyde resin plant.

World News

GREAT BRITAIN

Europe's surplus chemicals

A revised edition of the booklet entitled 'Chemical Products Exportable Surpluses,' has been published by the Organisation for European Economic Co-operation. The booklet is printed in English and French, and contains details about chemical products which other countries, including the United Kingdom, have available for export.

Only a limited number of copies is available, and supplies have been sent only to the undermentioned trade associations. Enquiries about the booklet and its contents should be addressed to the associations, who are prepared to make the information available to any firm in the chemical industry, whether a member or not:

The Association of British Chemical Manufacturers, British Plastics Federation, Association of Tar Distillers, National Benzole Association (N.B.A.) Ltd., Association of British Pharmaceutical Industry, Federation of Gelatine & Glue Manufacturers, the Export Group of the Paint Industry, the Printing Ink Export Group and the British Chemical & Dyestuffs Traders Association Ltd.

Private trade in chrome ore

Centralised purchase and sale of chrome ore ceased on November 21. Announcing this, the Ministry of Supply stated that import of chrome ore is now subject to import licensing regulations. For the past ten years the Government has been the sole importer and the sole supplier to U.K. consumers.

Rising output of petroleum products

The remarkable increase in British manufacture of petroleum products is shown in figures recently published by the Petroleum Information Bureau, London.

Britain's largest blast furnace

The new No. 2 furnace of the Steel Co. of Wales at Margam was blown in on November 7. This furnace, having a hearth diameter of 25 ft. 9 in., is the largest in Great Britain and is rated to produce 1,000 tons/day of basic iron. It forms one of the group which will supply hot metal to the steel plant at the Abbey works, where the largest hot-strip mill in Europe is now being installed. The new furnace, like the other at Margam, was designed and built by Ashmore, Benson, Pease & Co. Ltd., Stockton-on-Tees.

Safety rules supplement

Supplement No. 1 to 'Model Safety Rules for Use in Chemical Works,' Part 1, third edition, published in 1947, has recently been issued by the Association of British Chemical Manufacturers.

It contains the changes made necessary by the Factories Act, 1948, together with a few minor changes and additions as the result of experience in use. The amendments are printed on separate gummed slips to allow of insertion in the blank pages provided for that purpose in the existing rules. Copies of the supplement are being sent gratis to all known purchasers of the original edition of Part 1. The amendments will be incorporated in due course in future editions.

Copies of the third edition of Part 1, including a copy of this first supplement, are still available from the Association office at 166 Piccadilly, London, W.1, price 7s. 6d. post free.

As is illustrated in the following table, output of petroleum products (given in tons) from both imported and indigenous crudes has been more than doubled in the 11 years from 1938-49:

Product	1938	1948	1949
Motor and aviation spirit	316,531	610,802	870,138
Industrial spirits	2,585	3,950	4,083
White spirit	27,181	43,028	47,080
Burning oil	121,036	110,508	119,963
Vaporising oil	6,337	10,337	8,005
Gas/diesel oil	377,537	907,229	1,192,212
Fuel oil	644,402	1,396,626	2,391,761
Refinery fuel consumption (ex own production)		337,244	388,325
Bitumen	612,609	523,227	554,759
Paraffin wax, scale and slack wax	11,843	14,124	14,810
Lubricating oil	157,805	365,831	375,956
Propane	100	1,187	1,249
Butane	1,500	13,851	19,692
Other petroleum gases	—	199	593
Miscellaneous products and loss	112,256	123,424	175,164
Total	2,391,722	4,461,567	6,163,790

Indigenous materials made available for distribution by the petroleum industry are given in tons in the following table:

Product	1938	1948	1949
Motor spirit (by low-temperature carbonisation)	2,170	2,846	1,944
Motor and aviation spirit (by hydrogenation)	141,000	107,218	129,648
Refined benzole	214,510	243,051	251,966

Tar Confederation officers

Officers of the British Tar Confederation for 1950-51 are: *President*, Col. H. C. Smith; *Hon. Treas.*, Mr. C. E. Carey; *Chairman of the Executive Board*, Sir Walter Benton Jones; *Vice-Chairmen of the Executive Board*, Mr. W. K. Hutchison and Major A. G. Saunders.

The executive board consists of ten representatives nominated by each of the Association of Tar Distillers, the British Association of Coke Oven Tar Producers and the Gas Council.

Anodising patents lapse

Following a settlement of a claim between the Minister of Supply and Alumilite & Alzak Ltd., two patents concerned with sulphuric acid anodising of aluminium, which were assigned to the Minister of Supply in February 1950, have been allowed to lapse.

The patents are: Brit. Pat. 474,609, 'Improvements in and relating to Colouring Treatment of Anodic Coatings of Aluminium and its Alloys,' and Brit. Pat. 476,161, 'Improvements in or relating to the Anodising of Aluminium and its Alloys.'

The inventions of these two patents have been available to industry in this country since April 30, 1950.

Process instrumentation lectures

Courses in process instrumentation have recently been initiated by the Department of Coal, Gas and Fuel Industries with Metallurgy at the University of Leeds. The course, which will ultimately be taken by all degree and post-graduate diploma students in the subjects of gas and chemical engineering, ceramics and process metallurgy, is to be given in two parts. The first part, which has commenced in the current session, is 'The Measurement of Process Variables' and deals with principles and methods of measuring such quantities as pressure, temperature, fluid flow, etc. The second part, 'Automatic Control,' will commence in the following session and will deal with the theory and practice of automatic controls as applied to the process industries.

These lecture courses, in common with other lecture courses in the university, are open to external students by arrangement with the Department of Extra-Mural Studies.

Pipette standard

Sixteen sizes of pipette from 1 to 100 ml. capacity are specified in a new British Standard,* being those most in demand for general laboratory use. Full dimensions are given, but in accordance with current practice in British Standards for laboratory glassware, only the essential ones are listed as mandatory, the remainder being given

*Copies of B.S. 1583:1950 one-mark bulb pipettes may be obtained from the British Standards Institution, 24 Victoria Street, London, S.W.1., price 2s. post free.

for the guidance of manufacturers. Standard methods are included for the determination of capacity and delivery time, and tolerances listed for both. An appendix gives the sizes of glass tubing recommended for the manufacture of these pipettes.

GERMANY

Allies' plans for I.G. Farben

The Allies have informed the West German Economics Minister that their plan for reorganising I.G. Farbenindustrie would split it up into nine separate undertakings and not three as proposed by the German experts. These nine works are Ludwigshafen, Hoechst, Mainkur, Titan at Leverkusen, Filmwerke at Leverkusen, other works at Leverkusen, Dormagen Bottweil and Bobingen Werke grouped together and Huesland Rheinfelden.

The German reaction is that the Allied proposals will go some way towards meeting their views, but they will probably put up amendments to the scheme which the Allied experts will consider. Allied circles said there is no chance of the High Commission accepting the German plan for three groups of companies.

One of the German suggestions is likely to be that the artificial silk works should not be detached from the chemical works which provide their raw materials.

Oil processing projects

The Ruhrchemie A.G. at Oberhausen-Holten, formerly engaged in processing Fischer-Tropsch products, and the Omnipetrol Oil Processing Co. at Karlsruhe have concluded a contract providing for close co-operation in oil processing, the West German Economic News Agency, VWD, reports. Omnipetrol represents the West German interests of the Omnium Français Pétroles, Paris.

The contract, which provides for repair of the war-damaged Fischer-Tropsch installations of the Ruhrchemie and their use for crude oil processing and the construction of an additional refinery at Oberhausen, has already been approved by the Allied Security Board.

The crude oil supplies by Omnipetrol from the Middle East will be finished by the Ruhrchemie. A processing capacity of 200,000 tons is planned. The oil will be topped in a distillation plant, and residues of about 50% will be processed in a cracking plant. Diesel oil, heating oil and probably also refinery gas will be produced in addition to petrol. Petrol output was estimated at 100,000 tons p.a.

Repair work as well as construction of the new plant will be done by the Ruhrchemie in co-operation with German, French and American firms. The cost was estimated at about 10,000,000 DM.

Dutch to build paint factory

The management of the paint factory 'De Rem' at Groningen has decided to establish a second factory at Bremen-Hemelingen in Germany; this decision has been taken in view of existing difficulties with regard to the import and export of paints from and to Germany.

No details are disclosed about the production capacity of the new factory, which will have a Dutch management, but it will be equipped with the most up-to-date machinery.

SWEDEN

Trade with Poland

Chemicals, coal, iron and salt are included in the goods which Poland will export to Sweden under a new trade agreement. Total value of Polish exports will be £17,000,000. A total of £13,500,000 worth of goods will be exported from Sweden to Poland, among which will be iron ore, cellulose and iron and steel.

NORWAY

£600,000 urea factory completed

Norsk Hydro has now completed its new factory at Heröya for the manufacture of urea. Full production has begun at the rate of 30 tons/day, equivalent to 10,000 tons p.a. Urea is a snow-white powder, which when heated together with formaldehyde produces solid plastic materials which can be shaped and coloured to make a great variety of articles. The urea can also be used for making various glues for use in the veneer and plywood industry. Norsk Hydro will also use the urea to produce a special fertiliser which, because of its high nitrogenous content, is particularly suited for export abroad to markets where freight charges are high. Urea can also be used as an animal fodder. Urea is produced from ammonia and carbonic acid, which Norsk Hydro manufactures in large quantities. Norsk Hydro is Norway's largest industrial concern, and produces 1,000,000 tons p.a. of nitrogenous fertilisers.

PORTUGAL

New paper mill

The E.C.A. has approved the allocation of $2,013,000 for the establishment of a new 'kraft' wood-pulp and paper mill, which will make Portugal self-sufficient in this commodity and should permit some exports. Total cost of the new mill is estimated at $9,580,000, of which $6,240,000 are for equipment. Purchases of equipment will be made in the U.S. ($4,070,000); Great Britain ($1,966,000); and Sweden and Finland ($200,000). The Portuguese Government's participation in the project will be 50%.

As already stated in INTERNATIONAL CHEMICAL ENGINEERING (1950, January, p. 42, and May, p. 235), the mill will be located at Cacia. It is due to begin operations in 1952 with a production of 20,000 tons p.a. An annual production of up to 40,000 tons of pulp, half of which would be bleached, and 28,000 tons of paper will be attained later. Imports of pulp are currently estimated at 14,000 tons p.a.

SOUTH AFRICA

Paper mill planned

It has been decided to establish a new pulp and paper mill in Natal at an estimated cost of £5,750,000. Preliminary investigations have been carried out by the Union Corporation Ltd. and other concerns, and it is proposed that the new venture be taken over by S. African Pulp & Paper Industries Ltd., subject to shareholders granting the directors the necessary increased borrowing powers and increasing the capital of the company.

INDIA

Raw material shortages in plastics industry

Difficulty in obtaining essential raw materials is hampering the expansion of India's plastics industry. Polystyrene moulding powder, which is imported from Canada and the U.S., has been scarce since the outbreak of the Korean war.

The India plastics industry, which is of war-time growth, has expanded rapidly and has today 100 major factories, mainly around Bombay and Calcutta, and a capital of Rs.60,000,000 invested in it. The annual production of plastic goods is estimated at 3,500 tons and consumption at 6,000 tons. India has not imported any general plastic goods since the second half of 1949, in an effort to conserve her foreign exchange for essential needs. Electrical accessories are the sole plastic goods now allowed to be imported.

The industry is almost totally dependent on foreign sources for its essential raw materials, mainly the U.S. and U.K. Only one essential raw material, phenol formaldehyde moulding powder, is manufactured in India, and only partially meets the needs of the industry.

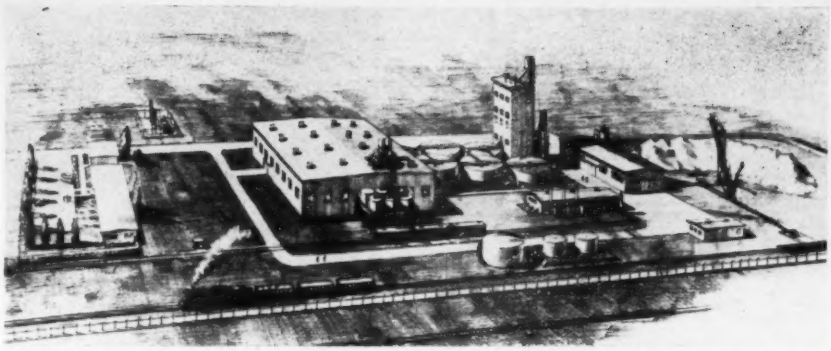
The annual consuming capacity of the industry of essential raw materials is estimated at 4,000 tons of thermoplastic raw materials, 3,000 tons of thermosetting raw materials and 250 tons of fabricating raw materials. The industry is suffering from lack of suitable trained personnel.

Research in plastics is being carried out under the auspices of the Council of Scientific and Industrial Research, a Government-sponsored body. The Bombay University's Department of Chemical Technology also gives courses on plastics.

Rise in output of heavy chemicals

Figures now available show a steady increase in production of heavy chemicals in India in 1950. Output of soda ash rose from 11,459 tons in the first quarter of 1950 to 12,207 in the second quarter; caustic soda from 2,550 to 2,720 tons; liquid chlorine from 998 tons to 1,154 tons; and sulphuric acid from 22,163 to 26,444 tons.

Regular manufacture of soda ash, one of the most important industrial raw materials, was undertaken in India in 1940. Since then the industry has made rapid progress.



Filtrol Corporation of Los Angeles will soon complete what is claimed to be the world's largest single plant for the manufacture of high-grade catalytic cracking catalysts. This new plant (*drawing above*) **will be located in Salt Lake City, Utah, and will produce a new type low-priced catalyst suitable for the production of high-octane petrol from cracking stocks of any sulphur content. This plant will make available much-needed catalyst for fill and operating make-up for both fluid and Thermofor-type catalytic cracking units now under construction in England and the Continent.**

Production increased from 11,614 tons in 1940-41 to 21,689 tons in 1947-48 and to 30,057 tons in 1949-50. Only two factories are at present producing soda ash. One was set up in 1940 and the other in 1944.

The annual demand for soda ash in the next two or three years is estimated at 90,000 tons. Installed capacity of the industry is about 46,800 tons. A considerable proportion of the demand is met by imports, the chief sources being the U.K., East Africa, the U.S.A., Italy, France and Japan.

New achievements in chemical research

Notable progress in chemical and drug research has been made at the Indian Institute of Science, Bangalore, according to a recently published report.

Work on the solvent extraction of vegetable oils is in progress. Extraction of castor seeds and cake with cheap solvents and the economic possibilities of the process in India are being studied.

Studies on catalytic cracking of petroleum products (kerosene) are being carried out to obtain gas with a high calorific value at lower temperatures (600 to 800°C.). The object of this study is mainly to reduce the consumption of coal as used in gas-house practice where cracking is done at higher temperatures (1,000°C.).

A new method of preparing the anti-malarial drug paludrine (discovered and developed in England by I.C.I.) has been discovered at the Institute and has been patented. Work has been started on other new methods of synthesising paludrine. A large number of compounds on the model of paludrine and a compound belonging to some of the other well-known anti-malarial drugs have been synthesised and are being tested pharmacologically.

Synthesis of new compounds derived from salicylic acid which has gained considerable importance in the chemotherapy of tuberculosis, is being carried out in the hope of finding a more effective agent for combating this disease. Work has also been started at the Institute on the preparation of new anti-tubercular and anti-leprotic compounds. Work has been carried out on synthesis of local anaesthetics of the novocaine type.

Coal for synthetic oil manufacture

Investigations recently conducted by Indian geologists have shown that adequate supplies of low-grade coal suitable for the manufacture of synthetic oil are available in the country. A survey was carried out in four coal-bearing areas in West Bengal and Bihar. It has been estimated that nearly 600,000,000 tons of quarry coal with an ash content varying between 12 and 25% could be obtained for this purpose.

The geologists have recommended establishment of a synthetic oil plant in the East Raniganj area in West Bengal. In the other three regions similar plants may be constructed at various coalfields after further investigations.

Germans to make drugs

The German Byk-Gulden Co. has concluded an agreement with the Biva Co. in Calcutta for the manufacture of cardiac remedies in India. It is stated that the agreement specifies complete Indian control of the organisation within ten years and the training of Indians in the best German methods and 'know-how.'

Pulps for rayon manufacture

New sources of high-grade pulps suitable for rayon manufacture have been brought to light as a result of the recent experiments conducted at the Cotton Technological Laboratory, Bombay.

Raw materials selected for investigation were certain varieties of bamboo reeds; bagasse and jute materials had alpha-cellulose of over 80% and ash contents varying from 0.2 to 0.3%. This meets the requirements for pulps used in the rayon industry.

CANADA

German gypsum plant ordered

The Newfoundland Government has ordered machinery for two gypsum mills from six German firms.

Vermiculite deposits

Canada's first commercially-usable deposit of vermiculite has been discovered at Stanleyville, Ontario, the Mines Department announces. A field investigation by the Industrial Minerals Division of the Department revealed the deposit in the area about 60 miles south-west of Ottawa. Its extent is not yet known.

The Department said preliminary tests indicated the product was of high quality and formed a valuable addition to Canada's known resources of industrial minerals.

Canada now imports about $250,000 worth of vermiculite a year, chiefly from S. Africa and the U.S., and Mines Department officials said the market for it was steadily increasing.

Vermiculite is used for insulating buildings, refrigerators, ships, aeroplanes and other vehicles; in making lightweight concretes and plasters and in paints and pigments.

AUSTRALIA

Tenders for oxygen separation plants

Details of a call for tenders, issued by the Department of Public Works, Melbourne, for the supply, erection, testing and putting into operation of oxygen separation plants were given in INTERNATIONAL CHEMICAL ENGINEERING, 1950, **31** (11), p. 523.

The U.K. Trade Commissioner at Melbourne now reports that the closing date for the submission of tenders has been extended to Tuesday, January 30, 1951.

BRAZIL

Polystyrene plant

A new plant is to be constructed near Sao Paulo to produce about 3,000,000 lb. p.a. of polystyrene plastics by the chemical division of Koppers Co. Inc., U.S.A. The plant will be built and operated by a newly organised Brazilian company, Companhia Brasileira de Plastico 'Koppers.' Besides furnishing most of the equipment for the plant, Koppers will provide engineering and technical assistance and will supply styrene monomer, the principal raw material for polystyrene production.

ANGOLA

New factories

A new cement factory, a fibro-cement factory and a castor-oil plant are being constructed at Lobito. A starch factory is under construction at Nova Lisboa, a brewery is being erected at Luanda and new installations are being made at the Quizenga manganese mines.

U.S.A.

Synthetic rubber output increased

Government plants are now turning out synthetic rubber at a rate of 430,000 tons p.a., according to the Reconstruction Finance Corp. This compares with 220,000 tons p.a. in January.

In a report on efforts to put standby plants into operation and increase output to meet defence requirements, the R.F.C. vice-chairman said that the present rate of output would be pushed above 600,000 tons p.a. by the end of 1951. The production rate of 760,000 tons ordered by President Truman should be attained in the following spring, he declared.

New synthetic petrol plant successful, others planned

For the first time in the U.S., petrol has been produced from coal in commercial quantities. The U.S. Bureau of Mines recently disclosed that more than 225,000 gal. have already been produced at its $25,000,000 demonstration plant at Louisiana, Missouri.

Petrol production at the plant, which was set up to test the feasibility of establishing a synthetic oil industry in the U.S., has been proceeding at rates up to 9,000 gal./day. The Bureau, however, does not plan to maintain daily output because technical appraisal of the process, rather than large production, is the chief object of the tests.

The Bureau of Mines said that, if a synthetic oil industry were established, the U.S. would achieve independence of foreign sources of petroleum in the event of war and would be able to meet the country's normal needs for oil for centuries to come. Known available coal reserves in the U.S. are estimated to be sufficient to last 1,000 years even if the nation made all its oil from coal.

The petrol is made by the hydrogenation process, the addition of hydrogen to coal under high pressure and temperature. The coal is reduced to a semi-liquid state before the addition of hydrogen and then, in the presence of a catalyst, the coal can be converted to petrol, diesel oil, jet fuel and various other products.

Present production concentration at the plant is on petrol and, according to preliminary estimates, costs will be not much higher than for producing petrol from natural petroleum.

The Bureau of Mines also disclosed that it expects to begin petrol production next spring by the Fischer-Tropsch process.

A third method of making synthetic oil is the gas combustion process. It is believed by Bureau of Mines engineers to be their most efficient and economical way to date for making oil from the vast domestic deposits of oil shale that up to now have been too expensive to develop commercially. The process consists simply of a large retort, down which raw shale is poured against an upward current of hot gas that converts the shale into oil. The Bureau intends to make larger-scale studies to determine the commercial value of the process.

Alkyd resin plant

The Blaw-Knox Co. has been engaged by the Plaskon Division of the Libbey-Owens-Ford Glass Co. to design and construct a new alkyd resin production plant at Toledo, Ohio. The plant will employ new processing techniques recently developed in Plaskon's laboratories. It will be air-conditioned with accurate temperature control, and its design will feature provisions for efficient materials handling and allowance for future expansion. It is anticipated that the plant will be in production by March 1951.

Cortisone plant

Merck & Co. are to build a new production unit at a cost of several million dollars at its Cherokee plant near Danville, Pennsylvania. The plant will manufacture *Cortone* (cortisone), an anti-arthritic hormone used in treating rheumatoid arthritis, rheumatic fever and other diseases. The new facilities at Cherokee will more than double the company's capacity for making *Cortone*. They are expected to come into operation late in 1952.

Zinc oxide output rises

Production of zinc oxide in the U.S. during October rose 9% (compared with the previous month), to 20,576 short tons and was at the highest monthly rate since March 1950, according to the Bureau of Mines. Stocks of zinc oxide on October 31 were 10% above the September 30 total, largely the result of a 45% gain in stocks of leaded zinc oxide.

Output of leaded zinc oxide rose 10% to 6,419 tons, and production of lead-free oxide was 14,157 tons, or 8% above September.

More ammonium bicarbonate

The Solvay process division of the Allied Chemical & Dye Corporation is to spend $2,500,000 on increasing the production capacity of its ammonium bicarbonate plant at Syracuse, New York. This programme is in addition to a $40,000,000 expansion project at the company's soda ash plant in the same area, work on which has already begun.

MEXICO

New aluminium plant

A large aluminium plant is to be established by Reynolds Metals Co., according to Mr. T. Hall Barnes, vice-president and manager of Reynolds Internacional. Mr. Barnes, who is at present touring Reynolds' plants in the U.S. in company with a group of Mexican officials and industrialists, added that the plans envisaged the setting up of the plant in the petroleum fields just south of the U.S./Mexican border. The plant, which would probably use pig aluminium from Arkansas, would fabricate material for the utensil industry.

Another member of the group, Sr. Antonio C. Flores, director-general of Mexican National Finances, disclosed that the plant would produce 40,000,000 lb. of fabricating material p.a. The project would require an investment of about $20,000,000. He and other officials of the Mexican Government and of the Banco Nacional de Mexico were visiting U.S. Reynolds' plants, he added, because the establishment of a factory in Mexico would require financial transactions with the Mexican Government's international banking representatives.

EL SALVADOR

First cement plant

El Salvador's first cement plant, costing $2,000,000, is under construction near Acajutla. It is being jointly financed by U.S. and Salvadorean interests. Machinery is being supplied by Allis Chalmers, Milwaukee, and building construction is being carried out by the MacDonald Engineering Co., Chicago. It has been estimated that the factory will produce 900 barrels/day of cement, more than enough to cover El Salvador's present consumption.

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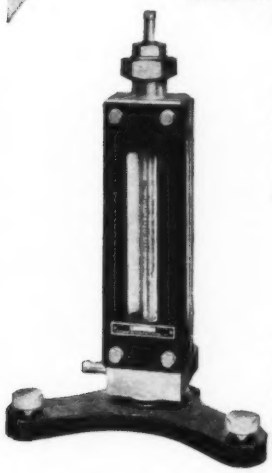

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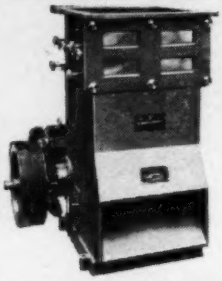

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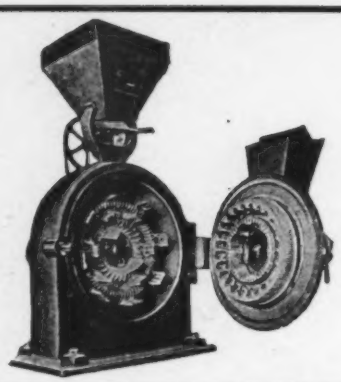

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